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IN ALL ITS APPLICATIONS TO

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ON THE VISCOSITY OF GASES AS AFFECTED BY TEMPERATURE.*

By Lord RAYLEIGH, F.R.S.

A FORMER paper (*Roy. Soc. Proc.*, 1900, lxvi., p. 68; *CHEM. NEWS*, lxxx., p. 85) describes the apparatus by which I examined the influence of temperature upon the viscosity of argon and other gases. I have recently had the opportunity of testing, in the same way, an interesting sample of gas prepared by Professor Dewar, being the residue, uncondensed by liquid hydrogen, from a large quantity collected at the Bath springs. As was to be expected (*Roy. Soc. Proc.*, 1896, lix., p. 207; 1896, lx., p. 56), it consists mainly of helium, as is evidenced by its spectrum when rendered luminous in a vacuum tube. A line, not visible from another helium tube, approximately in the position of D₂ (Neon) is also apparent.†

The result of the comparison of viscosities at about 100° C., and at the temperature of the room, was to show that the temperature effect was the same as for hydrogen.

In the former paper the results were reduced so as to show to what power (n) of the absolute temperature the viscosity was proportional.

	n.	c.
Air.. .. .	0.754	111.3
Oxygen.. .. .	0.782	128.2
Hydrogen }	0.681	72.2
Helium	0.815	150.2

Since practically only two points on the temperature curve were examined, the numbers obtained were of course of no avail to determine whether or no any power of the temperature was adequate to represent the complete curve. The question of the dependence of viscosity upon temperature has been studied by Sutherland (*Phil. Mag.*, 1893, xxxvi., p. 507), on the basis of a theoretical argument which, if not absolutely vigorous, is still entitled to considerable weight. He deduces from a special form of the kinetic theory as the function of temperature to which the viscosity is proportional—

$$\frac{\theta_1}{1 + c/\theta} \dots \dots \dots (x).$$

* A Paper read before the Royal Society, June 21, 1900.

† I speak doubtfully, because to my eye the interval from D₂ to D₃ (helium) appeared about equal to that between D₂ and the line in question, whereas, according to the measurements of Ramsay and Travers (*Roy. Soc. Proc.*, 1898, lxiii., p. 438), the wave-lengths are—

D ₁	5895.0
D ₂	5889.0
D ₃	5875.9
D ₄	5849.6

so that the above-mentioned intervals would be as 19.1:26.3. I may record that the refractivity of the gas now under discussion is 0.132 relatively to air.

c being some constant proper to the particular gas. The simple law θ_1 , appropriate to "hard spheres," here appears as the limiting form when θ is very great. In this case, the collisions are sensibly uninfluenced by the molecular forces, which may act at distances exceeding that of impact. When, on the other hand, the temperature and the molecular velocities are lower, the mutual attraction of molecules which pass near one another increases the number of collisions, much as if the diameter of the spheres was increased. Sutherland finds a very good agreement between this formula (x) and the observations of Holman and others upon various gases.

If the law be assumed, my observations suffice to determine the values of c. They are shown in the table, and they agree well with the numbers for air and oxygen calculated by Sutherland from observations of Obermayer.

THE SENSITIVENESS OF SILVER AND OF SOME OTHER METALS TO LIGHT.*

By Major-General J. WATERHOUSE, I.S.C.,
(late Assistant Surveyor-General of India).

DURING some recent investigations on the Daguerreotype process, the question presented itself as to which of the elements forming the sensitive surface of the plate—the silver or the halogens—the sensitiveness was due? Now, although the fact that nearly all compounds of silver, especially the haloids, are more or less sensitive to, and decomposed by, the action of light, has long been known, the sensitiveness of metallic silver itself to light, though observed in 1842 by Moser, has never been generally recognised either by chemists or by photographers.

Moser's Experiment.—Before describing my own experiments, it may be as well to give a description of Moser's experiment taken from the original paper in *Poggendorff's Annalen*, 1842, vol. lvi., p. 220.

"A perfectly new silver plate was thoroughly cleaned and polished. A black tablet with various excised characters was fixed above it, without touching it, and the whole placed in the sun for two hours or more and directed towards it. After the plate, which naturally did not show the least change, was cooled, it was held over mercury, heated as usual to about 60° R. (167° F.). To my great delight a distinct image of the screen was produced in which those parts where the sunlight (which during the course of the experiments was always weak and changeable) had acted had attracted a quantity of mercury. This interesting experiment was repeated

* A Paper read before the Royal Society, May 31, 1900.

several times with the same result. Sometimes the plates after having been placed in the mercurial vapours were exposed to those of iodine and then placed in the sun, by which the images usually improved."

"If we compare this remarkable fact of the action of light upon surfaces of silver with the above mentioned phenomena produced by contact, we can no longer doubt that light acts on all bodies, modifying them so that they behave differently in condensing the vapours of mercury. A similar experiment was made with copper during unfavourable weather. The copper was not well polished, and, consequently, the image produced by the mercurial vapours was faint, although clearly visible. By exposing the plate to the vapour of iodine the image became stronger, and this method was found useful in experiments with copper. A plate of clean mirror-glass was exposed in the same way to light, and the action was as plain as on the silver, if the glass were afterwards breathed upon, the image remaining visible for a long time afterwards. We may therefore assume that light acts on all bodies, and its influence may be tested by all vapours that adhere to the substance or act chemically upon it."

Robert Hunt's Views.—Although Robert Hunt recorded these experiments in his "Researches on Light," he does not seem to have repeated them as regards the direct action of light upon metallic silver, but to have paid more attention to Moser's experiments on the images produced by contact or proximity of dissimilar substances, and his theory of invisible light, as well as the effects of heat. Hunt's own experiments were chiefly carried out on copper plates, and led him to attribute Moser's results to calorific or thermic radiations rather than to light. Knorr, Karsten, Grove, and others seem also to have investigated Moser's theories, but again without taking notice of the fact of images, either directly visible or developable, being produced on metallic silver by the direct action of light.

I have not been able to find a record of a visible action of light upon ordinary silver plate, though its occurrence should be well known to silversmiths.

Carey Lea's Observations.—Carey Lea found that the three forms of allotropic silver he obtained were all sensitive to light: A, the red soluble, and B, the dark brown or blue insoluble variety, becoming brown after some hours' exposure, while C, the golden coloured, became lighter by exposure (*Phil. Mag.*, 1891, Ser. 5, vol. xxxii., p. 337).

Electrolytically Deposited Silver.—In a series of electrolytic experiments made in Calcutta in 1892, I found that a golden-yellow or light olive-coloured deposit of silver on the cathode plate (silver or platinum) of a decomposition cell formed with two pure silver plates, as anode and cathode, or with a silver anode and platinum cathode in distilled water, through which a weak current was passed, was slightly sensitive to light, and became lighter in colour by exposure. This seems to confirm Carey Lea's observation, if my golden-yellow silver deposit was analogous to his C-product. My deposit being made by electrolysis of pure silver in fairly pure distilled water must have been nearly pure silver, with no traces of foreign substances beyond those contained in the silver or the water, unless there was a small amount of occluded hydrogen, which other experiments of the same series, but with a stronger current, showed might not be impossible.

Photo-electrical Observations in Calcutta.—In another series of observations on the electrical action of light upon silver made in Calcutta about the same time, and published in the *Journal of the Asiatic Society of Bengal*, Part II., No. 1, for 1893, I found that the action of bright sunlight on a plate of almost pure silver, as indicated by a very sensitive Rosenthal galvanometer, was to make the exposed half positive to the unexposed, or as zinc to copper, which would seem to point to some slight oxidising action. The observation, however, was a difficult one, not readily repeated with certainty, and no very definite result was obtained. The currents observed did not appear to be due to unequal heating of the two halves

of the plate, because the direct application of heat to the exposed side produced at once a clearly marked current in the opposite direction. This effect was always the same, and could be readily repeated.

In further observations of the same series, in which pairs of pure silver plates were partly immersed in distilled water or good tap water, one plate being exposed to light while the other was covered, as in Becquerel's electric actinometer, the current in nearly all cases, though small, was as above, the exposed plate being positive to the unexposed, as it generally was when the silver plates were placed in dilute sulphuric, nitric, phosphoric, or hydrochloric acids.

Confirmation of Moser's Observation.—Recent observations of the action of light upon silver, made in connection with the working of the Daguerreotype process, have fully confirmed Moser's observation quoted above, and shown that silver, when exposed under ordinary conditions, shows distinct sensitiveness to light, and that not only can an invisible developable image be obtained, as was done by Moser, but by prolonging the exposure printed-out impressions are produced which are clearly visible after exposure. The difficulty is to make sure of obtaining a surface of pure silver, free from the presence of condensed gases or other foreign matter which might affect the plate and give it a sensitive surface. The silvered glass plates used have generally been cleaned and polished with well-washed tripoli, and the metal plates in the same way, sometimes after being well cleaned with fine emery paper, and, in some cases, after making the metal red-hot.

Various Silver Surfaces Sensitive to Light.—Repeated observations with silver surfaces of different kinds, pure silver plates and foil, silver leaf on varnished glass, Daguerreotype plates and silvered glass, have shown that by an exposure in bright sunshine of about half-an-hour to one or two hours, under an ordinary black and white negative or a cut-out design, photographic images can be obtained, which are sometimes clearly visible after exposure, especially if it is at all prolonged.

Development of Images on Plain Silver Plates.—Whether visible or invisible, these images can be developed by the vapour of mercury, or by ordinary physical development with acid solutions of ferrous sulphate or pyrogallol acid, to which a little silver nitrate is added, as in the old wet collodion process. The images were also produced when the negatives or cut-out masks were separated from the silver surfaces by sheets of thin mica, which, as Dr. W. J. Russell, F.R.S., has shown, stops the action of vapours upon a sensitive gelatine dry plate, and no doubt would do so upon a silver plate. As a rule the mica itself makes no difference in the action of light, and is quite transparent to it, though, in some cases, it may exercise a slight retarding action. It was found, however, that when the silver surfaces were exposed to light in hydrocarbons, such as fluid paraffin, benzole, turpentine, &c., no action took place under the parts screened by mica, though the fluids were perfectly clear and colourless. Under ordinary conditions of exposure in a printing frame the mica appeared to exercise no special influence upon the plates, except at the cut edges, as will be noticed further on.

Effects of Pressure.—That the images were not produced by differences of pressure was proved by leaving the mica screen, with the cut-out black paper design at its back, in contact with a polished silvered glass plate in darkness for twenty-four hours. There was no visible image, but on development with mercury vapour the mercury was deposited fairly evenly all over the plate, except just where the outside edges of the mica and the edges of some initials cut in it had been in contact with the silver. Here there was very little deposit, and the edges appeared as dark lines on a white ground. There was no sign of the black paper design. The fact that the images were readily obtained from ordinary black and white photographic negatives is further evidence against the results being due to pressure.

First Experiment with Silver Leaf.—My first experiment was made on the 14th August last with a piece of silver leaf laid down on a plate of glass coated with varnish, and exposed in the sun for a short time under a plain cut-out cardboard screen. On developing with mercury vapour a faint image was visible, the mercury having deposited on the part exposed to light.

Next day the experiment was repeated. A similar plate was exposed under a black and white gelatine negative of some lace for half-an-hour in bright sunshine. On developing with mercury a very distinct, though faint, image of the lace pattern was produced, the mercury again depositing on the parts exposed to light, as in Moser's experiment.

Effect on Polished Silvered Glass.—Thinking that these results might be partly due to some action of light upon the varnish underlying the silver leaf, a piece of polished silvered glass was exposed under the same negative for about an hour. Again a distinct image was developed with the mercury, but partly positive and partly negative. A somewhat similar result was obtained on another plate developed with the ordinary acid iron and silver developer already noticed.

Effect on Pure Silver Plate.—The next experiment was with a piece of nearly pure silver plate, carefully cleaned with "Globe" polish, and washed with benzene to remove all greasiness. After half-an-hour's exposure in dull sunlight and development with mercury, an image was produced similar to the two first on silver leaf, *i.e.*, the mercury had deposited upon the exposed parts.

These experiments with three different forms of silver surfaces and two methods of development, all giving results similar to those obtained by Moser, seemed at any rate to prove the correctness of his observation, and the sensitiveness of ordinary forms of silver to light.

With the exception of the first, the foregoing trials were all made with a black and white negative on glass. Others were then made with cut-out black paper screens and with like results.

Observation of Printed-Out Visible Image.—The first observation of an image visible on the silver surface after exposure and without any development was on a plate of nearly pure silver, cleaned with tripoli and ammonia, polished off with dry tripoli, and exposed on the 21st August for about half-an-hour in sunshine under a black paper cut-out screen. The parts exposed to light appeared lighter than the unexposed, but on development the mercury was deposited upon the unexposed parts. In this case the surface may have been affected by the ammonia used in cleaning, but it is also likely that the black paper used for the mask exercised some effect, as was afterwards found to be the case.

Another silver plate exposed for the same time under the same cut-out mask, but separated from it by a sheet of mica, did not show the visible image after exposure, though an image was readily developed with mercury vapour.

A day or two later, on the 24th August, the same experiment was repeated upon a plate of silvered glass exposed in the sun for half-an-hour under the cut-out mask, with a mica screen between it and the silvered surface. The image of the black paper design could be faintly but distinctly discerned in a suitable light, again appearing dark upon a lighter ground. Development with acid iron and silver brought out clearly the images of the paper mask, and of some letters cut out of the mica screen, as well as the edges of the mica screen itself.

A piece of silvered glass was then exposed for forty-five minutes under the same black and white negative as used in the first experiments, the silvered surface being protected from contact with the negative by a mica screen. In this case also a faint image was visible after exposure.

Several other prints, both from the lace negative and the paper masks, were made on silvered glass with longer

exposures, so as to obtain a distinctly visible image, and it was then found that there was a tendency to reversal of the image when developed with mercury vapour, *i.e.*, the mercury deposited upon the unexposed parts instead of upon the exposed. In one plate the image thus produced from the negative of lace, exposed for two hours in the bright August sunshine, has quite the appearance of an ordinary Daguerreotype picture on an iodised silver plate, though no halogen or other sensitiser was used.

Further trials of prolonged exposures of pure silver foil or plates, carefully cleaned with dry tripoli powder, have given very distinct printed-out images on the metal, so that there is no doubt about the fact that visible images can be produced on clean plain silver surfaces by light.

Blue Rays found to be Active.—In order to ascertain, if possible, what rays were active in producing these visible images upon the silver, and as it was hopeless to expect to obtain satisfactory results from observations with the solar spectrum, a slip of silvered glass was exposed under a small artificial spectrum of seven coloured glasses. After forty-five minutes' exposure in sunshine very faint images of the violet and cobalt blue glasses were distinguishable, but showed more distinctly when breathed upon. A similar result was obtained upon a pure silver plate, and on developing with acid iron and silver the space exposed under the cobalt blue glass developed out quite clearly, with traces of the violet and blue-green glasses. This result quite agrees with an observation made by Moser that only the blue and violet rays have any influence on pure silver, for he obtained very clear images by means of glasses of these colours, while only traces could be rendered visible when red glasses were employed, although they transmit more light and heat.

On another silvered glass plate, exposed under a similar colour screen or artificial spectrum, consisting of fifteen coloured glasses, for three hours in bright sunshine, the image has apparently reversed by over-exposure, the mercury being deposited on the spaces exposed under the red, orange, yellow, and yellow-green glasses, but not on those which were under the blue-green, blue, and violet glasses.

As we have seen, Robert Hunt was inclined to attribute Moser's results to the effect of heat or differences of relative temperature rather than to light or solar radiations, but his experiments were mostly carried out on copper, which is, as I have found myself, much more sensitive to rays of low refrangibility and to heat than silver is.

Developable Images Produced on Silver by Heat.—Towards the end of September, when the weather was much cooler than it had been at the commencement of my experiments in August, I found that there was a distinct falling off in the sensitiveness of the silver surfaces, and it seemed that Hunt's view might, at any rate to some extent, be correct. I therefore tried an experiment to see if the same developable images could be obtained by heat as by light. A silvered glass plate was polished and put into a printing frame with the cut-out paper mask and mica screen, in which were cut-out initials, just as if it were going to be exposed in the sun, but it was gently warmed for about five minutes over a spirit-lamp, and then developed with mercury. The cut-out initials and edges of the mica came out distinctly in dark lines, just as they did in the pressure experiment, but there was also a clear image of the black paper mask, which developed lighter than the ground, by the deposition of mercury, or the opposite of the ordinary action of light.

This is a very interesting observation, but recent repetitions of it with silvered glass plates and clean silver foil have quite failed to give such a distinct image of the black paper, though traces of it have been visible, and the edges of the mica and of the cut-out initials were always clearly impressed. In one case, when the silver foil was well heated to redness on both sides before being placed in the printing frame and the subsequent heating, no image of the initials was obtained, and only part of one edge of the

mica screen with a faint trace of one corner of the paper mask where there was extra pressure. From this it would seem that heat does not play any active part in the production of the images, though the higher temperature of the summer sunshine, as well as its greater actinic power, may accelerate their formation by light. This is, to a certain extent, proved by the fact that the most perfect printed-out image obtained on a pure silver plate was exposed for three days at the end of September, when the thermometer exposed in the sun at the same time did not rise above 64°F. , so that there could have been no question of heat producing the effect, as there might have been under the hot, clear sunshine of August.

Protection from Air.—In most of the experiments the plates were protected by glass during exposure, so that the outer air had no direct access to them. When plates were exposed under mica screens and without the protecting glass, the outside unprotected surface became distinctly yellow and tarnished with long exposures.

Under Surface of Silvered Glass Plates not Sensitive.—In order, however, to ascertain the effect of cutting off all atmospheric action on the exposed side, a silvered glass plate was exposed from the back or glass side under a cut-out mask made of thin aluminium sheet, and exposed for four days in October, two days being sunny and two cloudy. There was no visible image on either side of the plate after exposure, but breathing showed an image on both sides. The plate was then developed with acid iron and silver, and showed the image not very distinctly, but as development was prolonged traces of it appeared at the back of the silver film quite clear of deposit, so that apparently the developer had worked through the protected parts of the film, while the exposed part had taken the deposit of silver. When the plate was dry there was, curiously enough, no trace of the image on either side of the plate.

This experiment was repeated in January, two silvered glass plates being exposed face to face for fifteen days, of which five or six were sunny and the rest fairly bright, with the object of also seeing whether an impression could be made through the upper plate on to the silvered surface of the under one. On developing with mercury a fairly clear image of the cut-out design was obtained on the inner surface of the exposed plate, dark on a lighter ground, but neither on the outer exposed silver surface of the upper plate, nor on the silvered surface of the lower plate was there any trace of an image. The experiment was repeated with a similar result. The only other case in which images have been obtained through the silvered film was on a plate partly fumed with hydrogen peroxide and developed with mercury, but much overdone. As far as they go, the experiments show that the visible image is not produced on the silver except more or less in contact with the air. Further trials in bright sunny weather are required to prove this.

(To be continued).

THE CRYSTALLINE STRUCTURE OF METALS.* (SECOND PAPER.)

By J. A. EWING, F.R.S.,
Professor of Mechanism and Applied Mechanics in the University
of Cambridge,
and W. ROSENHAIN, B.A.,
1851 Exhibition Research Scholar, Melbourne University.

THE investigations described in this paper deal principally with the phenomena of annealing. The first section of the paper describes experiments made in the hope of observing under the microscope the process of re-crystallisation in strained iron. It is well known that re-arrangement of the crystalline structure of iron occurs when the metal is heated to redness, and it is believed

that such changes are associated with the evolutions of heat which are indicated by "arrest points" during the cooling of iron. We hoped that by keeping a polished and etched surface of the strained metal under microscopic observation while the specimen was gradually heated, we should see a more or less sudden change in the crystalline pattern at a temperature corresponding to one of the "arrest points." This attempt, however, to watch the process of re-crystallisation failed, although the experimental difficulties of keeping a specimen under microscopic observation while it was being heated were successfully overcome. The specimen was electrically heated in a vessel with a thin glass or mica window, and the microscope-objective was kept cool by directing a strong blast of cold air on it and on the surface of the window. In one set of experiments the specimen was kept in an atmosphere of pure hydrogen during the heating, but it was found to become so much tarnished as to obliterate the crystalline pattern. At a red heat, however, the uniform luminous surface of the specimen was seen to develop a number of dark patches which, on slightly raising the temperature, spread over the entire field. No corresponding change was visible during cooling, but the phenomenon would recur every time the specimen was heated, provided it had been cooled below redness after the previous heating. This phenomenon was absent when the specimen was heated in a vacuum, and we believe that it indicates a chemical action between hydrogen and iron, possibly corresponding to the hydrogen arrest point discovered by Sir W. Roberts-Austen.[†]

In the next series of experiments the specimen was heated in a vacuum. On prolonged heating the specimen still became tarnished, but at first the crystalline pattern remained visible up to a bright red heat. No change in the pattern was observed, but subsequent polishing and etching of the same surface showed that a real change of crystalline structure had occurred. The original etched pattern on the surface had persisted after heating, simply because the differences of level and surface texture on which it depended had in no way been disturbed by the re-crystallisation. Any crystalline pattern seen under the microscope, whether it be produced by etching or relief polishing, consists either of coloured surface deposits, or of steps, or pits, or other differences of level in the surface, and these differences of superficial texture, like mechanical scratches, are not affected by re-arrangement of the crystalline elements. Coloured surface deposits would also remain unaffected. All attempts to observe the actual process of re-crystallisation must therefore be unsuccessful.

The next section of the paper deals with the changes of crystalline structure which go on in lead and other metals at comparatively low temperatures. Our attention was directed to this by noticing that a piece of plumber's sheet lead, when etched with dilute nitric acid, exhibits a strikingly crystalline structure, with large crystals. The character of this appearance led us to the view that a slow process of annealing or re-crystallisation was at work in such lead at ordinary atmospheric temperatures, and we have satisfied ourselves that this is the case. The method of investigation consisted in taking a series of micro-photographs, at low magnifications, of certain marked areas in the surface of a specimen, in order to watch the change which went on through lapse of time, or after application of some thermal treatment. It was necessary, for the reasons given above, to re-etch the surface before each photograph was taken.

We have observed that when a piece of cast lead is severely strained by compression, the originally large crystals, after being considerably flattened, are driven into and through one another, so that the etched surface of a strained specimen presents a fine grain, whose crystalline nature only becomes apparent under considerable magnification (30 to 100 diameters). A piece of lead severely

* Abstract of a Paper read before the Royal Society, May 31, 1900.

† Alloys Research Committee Report (*Proc. Inst. Mech. Eng.*), 1899.

strained in this way, and kept for nearly six months in an ordinary room without any special thermal treatment, was found to be undergoing continuous change during that time. A series of photographs of this specimen, taken at intervals during the six months, show that a great number of the small crystals have grown larger at the expense of their neighbours. In similar specimens which have been kept at 200° C., the growth has been much more rapid and more pronounced. The rate of growth is a function of time and temperature, but some specimens show much more rapid changes than others under similar conditions of temperature; in some cases five minutes' exposure to a temperature of 205° C. is sufficient to alter the crystalline pattern completely. Experiments have also been made at 105° C. and 150° C., leading to the general result that crystalline growth will occur at any temperature from that of an ordinary room, *i. e.*, 15° C. or 20° C., up to the melting-point of lead, and that in general the higher the temperature the more rapid is the initial rate of change. No numerical data can be given, as the crystals are quite irregular, both in size and shape. So far as our observations go, they lead to the result that when such crystalline growth has continued for some time at a given temperature, the structure becomes more or less stable, so far as that temperature is concerned, but exposure to a higher temperature may cause further growth to occur.

A comparison of micro-photographs of the same specimen at various stages reveals the fact that the growth of an individual crystal occurs, not in uniform layers all round it, but by the formation of arms and branches that invade the neighbouring crystals, the intervening portions sometimes changing at a later stage. This action is analogous to the formation of skeleton crystals in a metal during solidification from the liquid state, the space between the branches filling in as solidification proceeds.

A marked feature observed in several specimens was the large and rapid growth of one or two individual crystals; in several instances such individuals grew until they were some hundreds of times larger than their neighbours. We have not been able to discover the determining cause of such growth, nor, in general, why one crystal should grow at the expense of its neighbour. Generally the most aggressive crystals were found near the edges of the specimen. It is noticeable that at times a crystal which has already grown considerably is swallowed up by a more powerful neighbour.

Some light is thrown on the nature of these actions by the fact that this growth only occurs in crystals that have been subjected to severe plastic strain. By casting the metal in a chill mould, specimens of lead can be obtained having a crystalline structure quite as minute as that found in a severely strained specimen, but this structure remains unchanged at temperatures which produce rapid change in a strained specimen.

The investigation of the effects of such comparatively moderate temperatures was extended to other metals, *viz.*, tin, zinc, and cadmium. In tin, the various phenomena of crystallisation from the fluid state are strikingly illustrated on a large scale by the thin layer of that metal which constitutes the surface of commercial tin-plate. The effects of rapid and slow solidification in producing small or large crystals respectively are well marked, and an examination of the etched surface of tin-plate under the microscope reveals beautiful geometrical markings or pits, whose orientated facets produce the well-known selective effect of oblique examination. The study of the crystalline structure affords an explanation of the nature and method of production of patterns in "*moirée métallique*," a process which has long been in use for the decoration of articles manufactured of tin-plate.

In tin, also, we find that the smallest structure obtainable by quenching the melted metal in water remains unchanged at all temperatures up to the melting-point; on the other hand, specimens whose crystalline structure has been modified by great plastic strain exhibit phe-

nomens of re-crystallisation at lower temperatures similar to those observed in lead. In a piece of strained tin, an hour's exposure to 150° C. produced complete re-crystallisation. Exposure to lower temperatures for this short time produced no visible change, but we have not investigated gradual time effects in this metal. The behaviour of strained zinc and cadmium is analogous to that of tin and lead. Exposure to 200° C. is sufficient to produce rapid re-crystallisation in both zinc and cadmium. This is particularly marked in the case of ordinary sheet zinc. On etching commercial sheet zinc, no large crystals are visible. In this state the metal is strong and tough, bending quite noiselessly. After exposure to 200° C. for half-an-hour, it shows on etching a large brilliant structure, and the metal is then weak and brittle, and, when bent, breaks along well-marked cleavage planes and emits a "cry" like that of tin.

In cadmium the re-crystallisation is comparatively slow at 200° C., and a time effect has been observed; the action is rather different from that observed in lead. In cadmium the size to which the crystals grow appears to be much more uniform; no arms or branches are thrown out and no twin lamellæ are found.

The final section of the paper deals with an hypothesis, which is advanced as an attempt to explain the mechanism of the growth of crystals in apparently solid metal.* According to this hypothesis, the metallic impurities which are present in a metal play an important part in the action. When a metal solidifies from the fluid state, the metallic impurities ultimately crystallise as a film of eutectic alloy in the inter-crystalline junctions; when fairly large quantities of such eutectics are present, the microscope reveals their presence as an inter-crystalline cement, such as that formed by "pearlite" in slowly cooled mild steel; very minute quantities of eutectic, however, will be invisible and yet capable of forming a thin film of fusible cement. We conceive that the changes of crystalline structure which go on while the piece is in the solid state are accomplished by the agency of eutectic films between the crystals, in dissolving metal from the surfaces of some crystals and depositing it on others. When a metal is severely strained, these films of eutectic will be also strained and in many places broken, thus allowing the actual crystals to come into contact with one another. The difference in the rate of etching of adjacent crystals and the phenomena of the electrolytic transfer, in an acid solution, of lead from one crystal to another in the same mass of metal, support the supposition that there is a difference of electric potential between the crystal faces which are brought into contact by severe strain. If it be assumed that a film of eutectic alloy when fluid, or even when in the pasty condition that precedes fusion, can act as an electrolyte, we may regard any two crystals thus in contact, with a film of eutectic interposed in places, as a very low-resistance circuit, and the growth of the positive crystal at the expense of the negative would result. Moreover, such growth would be more rapid at higher temperatures, and its rate at a given temperature would vary in different specimens according to the nature and quantity of the impurities present. That an alloy can act as an electrolyte has not been established experimentally, but the assumption is supported by the close general analogy between alloys and salt solutions. This analogy extends to the very question of the growth of crystals, as Joly has shown that when crystals of a salt are immersed in their mother-liquor, growth of one at the expense of others will take place.

It should be added that solution of one crystal into the intervening film of eutectic, along with deposit on the neighbouring crystal from the eutectic, may occur as a consequence of differences of orientation, producing differences of "solution pressure" apart from actual electrolysis, but the fact that growth has not been ob-

* It is proper to say that this hypothesis is due to Mr. Rosenhain, —J. A. E.

served to occur except in strained crystals favours the view that the action is electrolytic.

Some further results which have been deduced from the above hypothesis have been verified by experiment. It follows from the hypothesis that an inter-crystalline boundary containing no eutectic would be an impassable barrier to crystalline growth, but if the eutectic could in any way be supplied, growth across the boundary might take place. In an absolutely pure specimen of lead there would be no eutectic at the inter-crystalline junctions, but as extremely minute traces of impurity would suffice to set up the action it is almost hopeless to verify the hypothesis in this way. Some experiments on the cold welding of lead have, however, borne out our conclusions. Two clean, freshly scraped, lead surfaces will unite under great pressure in the cold state, and if a piece so welded be annealed, we find that the crystalline growth due to the annealing, with very rare exceptions, never crosses the inter-crystalline boundary formed by the welding surface. To test whether the presence of some eutectic would allow growth to take place, we have scattered small quantities of a more fusible metal over the freshly scraped surfaces of lead before squeezing them together. Then, after a cold weld had been made by pressure, on annealing by exposure to 200° C. it was found that crystal growths frequently crossed the line of the weld, as the above theory led us to expect. This experiment has been repeated many times with the uniform result that whenever a small quantity of eutectic, or of an impurity capable of forming a eutectic with the lead, was scattered over the clean surfaces before welding a distinct growth of crystals across the boundary took place as a result of annealing. On the other hand, a large number of welds were made without introducing any impurity, and with very rare exceptions they showed no growth across the boundary, even after the annealing process was continued for some weeks. In rare exceptions a minute amount of growth across the boundary was observed, but these may fairly be accounted for by the almost unavoidable presence of traces of impurity. The result as a whole goes far to confirm this solution theory of crystalline growth in annealing.

THE COMPOSITION OF SOOT FROM MINERAL COAL.

By H. WARTH, D.Sc.

THE soot which is removed in large quantity from the chimneys of ordinary fire-places in which coal is burnt appears to be, at least in Birmingham, quite a commercial article on account of its value as a fertiliser. This made me enquire about the proportion of ammonium salts which it might contain. On turning to a chemical handbook, I found it stated that no data are available as to the composition of soot from mineral coal. I therefore made the examination following:—I selected a sample of the soot which had been collected from an ordinary household chimney under which coal had been burnt, said to have come from Cannock and Rugeley colliery, Hednesford. The sample weighed 368 grms. I first extracted the soluble matter by boiling with water, filtering, and evaporation. The product of evaporation was strongly hygroscopic, and in order to purify it still further I sublimated it, so as to obtain the ammonium salts separately as sublimate. The remainder was lixiviated with water and filtered, so as to obtain the soluble fixed (non-volatile) salts separated from carbon, &c.

The following quantities were obtained:—

	Percentage of salts from the original soot.			
Ammonium sulphate	..	..	..	0.1
Ammonium chloride	..	..	..	7.3
Total ammonium salts	..	..	..	7.4
Total soluble fixed salts ..	..	..	..	1.3

These fixed salts consisted of sulphates and chlorides of sodium, magnesium, calcium, and iron. The fixed salts contained much more sulphate than chloride. The proportion may have been about three of the former to one of the latter. This explains the almost total absence of sulphate amongst the volatile portion. The sulphur trioxide was chiefly retained by the non-volatile metals, and thus it is that the volatile portion consists of nearly pure ammonium chloride. The proportion of ammonium salt in the soot is large enough to justify the estimation in which the soot is held as a plant manure.

Birmingham, June 22nd, 1900.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART III.

By HARRY BREARLEY.

(Continued from vol. lxxxi., p. 77).

CARBON.

PAPERS on the relation of carbon to iron, its mode of existence after various kinds of physical treatment, &c., have been almost entirely neglected.

The processes for estimating CO₂ in various solid, liquid, and gaseous compounds and mixtures have also been neglected, either because they are more properly treated in another section, or are of only second-rate interest in the steel works laboratory. For a like reason, a large number of papers on organic analysis—of which the estimation of carbon after its separation from iron is but a particular case—are not referred to. These omissions are not intended to suggest what may and what may not be neglected in an analyst's equipment. Some such omissions were, however, necessary in a compilation which aims at making each reference bear directly on the practice of steel works laboratories.

The following is the classification adopted:—

- I. DIRECT COMBUSTIONS.
 - a. Dry Way.
 - b. Wet Way.
- II. MODES OF RELEASING THE CARBON.
- III. ESTIMATING THE LIBERATED CARBON.
 - a. By Weighing the Dried Residue.
 - b. Dry Combustion of the Residue.
 - c. Wet Combustion of the Residue.
 - d. Volumetric Estimation of the Residue.
- IV. COLORIMETRIC ESTIMATION.
 - a. Eggertz Process and Modifications.
 - b. Tintometers and other Apparatus.
- V. OTHER PROCESSES OF ESTIMATING CARBON.
- VI. ESTIMATION OF GRAPHITE.
- VII. MISCELLANEOUS NOTES.

The journals referred to are the same as in Part II., including the volumes completed in 1899.

I. DIRECT COMBUSTION OF THE SAMPLE.

a. In the Dry Way.—

307. MULDER (C. N., v., 5 and 210).—Cast-iron mixed with pumice stone is burned in oxygen. Regnault's mixture of KClO₃ and PbCrO₄ does not liberate chlorine.
308. HERMANN (C. N., xl., 263).—Direct combustion in O in a short platinum tube. The Fe₂O₃ formed verifies completeness of the combustion.
309. FÖRSTER (Z. I. S. I., 1895, ii., 588).—Ignite with PbCrO₄ in small porcelain retort. Large pieces of iron are oxidised more readily than very fine powder.
310. LORENZ (Z. C. S., lxiv., ii., 291; and lxvi., ii., 119).—Mixture of steel and PbCrO₄ ignited to white heat in oxygen.

311. LJUBAVIN (C. N., lvi., 221).— PbCrO_4 may evolve CO_2 on ignition; it should be fused in a Cu crucible.
312. PETERSSON (J. I. S. I., 1890, ii., 852).—Steel decomposed with KHSO_4 ; CO_2 and SO_2 absorbed in NaHO and $\text{Ba}(\text{HO})_2$; SO_2 oxidised to SO_3 , and CO_2 liberated with acid. Graphite is collected in a Pt funnel and burned in air and nitrous fumes. Later (J. I. S. I., 1893, ii., 527), SO_2 is retained by CrO_3 and gases passed over CuO into $\text{Ba}(\text{HO})_2$.
313. SCHNEIDER (J. I. S. I., 1894, ii., 488).—The filings are indirectly oxidised by igniting with metallic Pb or Cu. Carbide carbon is separated from iron with very dilute H_2SO_4 . Later (J. I. S. I., 1896, i., 531), purified phospho copper is used instead of powdered Cu.
314. BREARLEY and LEFFLER (C. N., lxxv., 241, 263, &c.).—Carbon in ferro-chromium can be estimated by igniting with PbCrO_4 , CuO , or PbO_2 . PbCrO_4 and CuO require high temperatures, PbO_2 does not.
315. ROZVETTI (J. S. C. I., 1899, 865).—Direct ignition of fine steel borings or powdered ferro-chromium with pure Al_2O_3 ; the process completed in thirty-five to ninety minutes.
316. SANITER (C. N., lxxv., 287 and 311).—Ignites with a mixture of litharge and CuO .
317. PARRY (J. I. S. I., 1880, 442).—Has always been able to detect CO in steel. (Would this heighten the results obtained by direct combustion?—H. B.).
318. FUNK (J. C. S., lxx., ii., 274).—The C in Zn is determined by direct combustion in oxygen.
- I.b. In the Wet Way.*—
319. JUPTNER (J. I. S. I., 1883, 779).—The sample is digested with CrO_3 and a large amount of H_2SO_4 . The evolved gases are dried and passed through KHO.
320. GMLIN (J. I. S. I., 1885, 246).—Very much like Juptner's process.
321. HEMPEL (J. I. S. I., 1895, i., 503).—Decompose sample with CrO_3 and H_2SO_4 in the presence of Hg; measure the CO_2 evolved, after exploding in a pipette to destroy hydrocarbons. No hydrocarbons are evolved on treating grey pig-iron.
322. WIBORG (J. S. C. I., 1887, 748; and 1890, 768).—Treat Fe with CuSO_4 , and, without filtering, oxidise C with $\text{CrO}_3 + \text{H}_2\text{SO}_4$; measure CO_2 in tube. The deposited Cu protects Fe from acid until a high temperature is reached: at lower temperatures hydrocarbons are evolved.
323. VON REIS (J. I. S. I., 1888, i., 376; and 1889, ii., 475).—Describes and commends Wiborg's process.
324. JUPTNER (J. C. S., lvi., 186).—Describes Wiborg's process.
325. LUNGE and MARCHLEWSKI (J. C. S., lxiv., ii., 45; lxxiv., ii., 188; and J. I. S. I., 1894, ii., 484).—Modified Wiborg process. H_2O_2 is used to generate O in the liquid, so as to expel CO_2 . Separation of Fe with Cu solution and wet combustion of residue inaccurate (see also J. C. S., lxii., 531).
326. DONATH and ERLHOFER (J. I. S. I., 1897, ii., 496).—A modification of Wiborg's process.
327. REINHARDT (J. I. S. I., 1892, ii., 511).—Gases burned to CO_2 by passing over electrically heated Pd spiral, and measured over Hg. Later (J. I. S. I., 1893, i., 403), uses heated platinated asbestos or prepared pumice.
328. BLAIR (J. C. S., lxx., ii., 544).—A modified Wiborg process. Combustion made with $\text{CrO}_3 + \text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$, and CO_2 measured over Hg.
329. ROUFF (J. S. C. I., 1899, 176).—Sample decomposed with CuSO_4 and a mixture of sulphuric, chromic, and phosphoric acids. The evolved CO_2 is measured. Not so good for ferro-alloys as for steels.

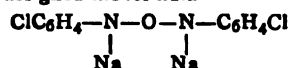
330. CAMPREDON (J. S. C. I., 1898, ii., 84).—Uses Wiborg's process after separating the C or graphite.
331. KOCH (J. S. C. I., 1894, 979).—Like Juptner, but passes gases over hot CuO . Special apparatus illustrated. (See also WUST, J. C. S., lxx., 449).
332. VON REIS (J. I. S. I., 1894, ii., 485).—Hydrocarbons are given off from direct combustion with CrO_3 and H_2SO_4 ; H_2SO_4 absorbs them; therefore dries with phosphoric anhydride instead. Addition of CuSO_4 lessens evolution of hydrocarbons. A constant error of 2 per cent is allowed for. (See also J. I. S. I., 1888, i., 374). Pumice moistened with KHO a better CO_2 absorbent than KHO bulb.
333. WOOD (J. S. C. I., 1884, 652).—Decompose sample with acid, pass gases through H_2SO_4 , alkaline Pb solution, and combustion tube into KHO.

(To be continued).

THE REDUCTION OF NITROBENZENE BY SODIUM.

By JULIUS SCHMIDT.

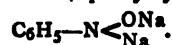
By reacting with metallic sodium on parachloronitrobenzene in ethereal solution, A. Hoffmann and A. Geyger obtained a very unstable black substance, to which, under reserve, they assigned the formula—



They did not succeed in preparing an analogous metallic compound with nitrobenzene, and they assume that sodium has no action on this nitrated hydrocarbide under these conditions. The author of this paper, who has repeated the experiments of the above-mentioned chemists, has found that, contrary to what they state, a reaction does occur at the ordinary temperature, between the sodium and the nitrobenzene, conforming to the following equation:—



The new compound which is thus formed may be regarded as a bisodic derivative of β -phenylhydroxylamine—



According to the author the reaction takes place much more energetically if, instead of operating at the ordinary temperature, the sodium is added to a boiling solution of nitrobenzene in toluene. The supposed bisodic substance occurs in the form of a blackish-brown powder possessed of a very considerable reducing power.

Decomposition of the Product of the Reaction under the Influence of Dilute HCl.—By treating the compound in question with double normal hydrochloric acid, a transformation takes place which gives rise to the formation of phenylhydroxylamine.

Slow Oxidation of the Product in the Air.—If we pass a current of dry air through cold ether containing the blackish-brown bisodic derivative in suspension, this latter gradually takes a brick-red colouration, and after the lapse of several hours is no longer capable of reducing an ammoniacal solution of silver, or Fehling's solution. If it is then treated with dilute H_2SO_4 , CO_2 is given off, and, by distillation of the acid liquor with steam, we obtain orthonitrophenol.

Oxidation of the Sodid Derivative with the Aid of Bichromate of Potassium and Sulphuric Acid.—By submitting the product to the action of this oxidising mixture, the author has obtained a compound, which he was able to recognise as nitrosobenzene, by its crystalline structure, its fusing-point (68°), and its odour.—*Berichte*, xxxii., p. 2911.

ON
CRYSTALLISED SELENIDE OF MANGANESE,
AND ON AN OXYSELENIDE.

By M. FONZES-DIACON.

FUSED selenide of manganese has been prepared by M. Fabre, by the action of selenium fumes on manganese heated to redness. This author has also obtained an amorphous reddish-grey selenide of manganese by the wet way.

I have prepared this body in a well crystallised form, both by the wet and dry way.

By the Wet Way.—Seleniuretted hydrogen precipitates a solution of acetate of manganese, giving, if the current passes slowly, a blackish-grey selenide, which is first deposited, followed by an orange-yellow selenide which forms a second layer, quite distinct from the first.

The blackish-grey selenide is crystalline, the yellow selenide is amorphous. If we add a small quantity of hydrochloric acid to the solution of acetate of manganese, and then saturate it with a current of seleniuretted hydrogen, the precipitation is not complete, but after two or three weeks a blackish-grey crystalline precipitate is formed, which under the microscope consists of beautiful non-modified cubes.

This selenide, dried in hydrogen to prevent its easy decomposition by air, has the formula $MnSe$.

Solutions that are too strongly acid are not precipitated by seleniuretted hydrogen; if the precipitation is carried out without proper precautions, we obtain a reddish-grey selenide of manganese, made up of a mixture of black crystalline selenide and orange-yellow amorphous selenide.

By the Dry Way.—Seleniuretted hydrogen reacts at a dull red heat on dried chloride of manganese giving crystallised selenide of manganese. The boats contain a black mass with a brilliant blown-out surface; the bubbles are lined on the inside with fine grey prismatic needles, appearing yellow by transmitted light. These crystals, which answer to the formula $MnSe$, are of too small a size for their crystalline form to be accurately determined.

We can also prepare crystalline selenide of manganese by reducing by carbon, at the high temperature of the electric furnace, a mixture made in the proportion of 1 molecule of seleniate of manganese to 4 molecules of sugar charcoal. This mixture, kept for ten minutes at the temperature of an electric arc of 80 volts and 140 ampères, becomes transformed into an ingot of selenide of manganese having a crystalline fracture with a brilliant grey appearance.

The action of this high temperature prolonged for even half-an-hour does not drive off any selenium.

We can also obtain a similar selenide by melting precipitated selenide of manganese in a carbon crucible, placed in a Moissan furnace in such a manner that the bottom part is heated. After heating for ten minutes with a current of 80 volts and 140 ampères, we obtain an ingot with a metallic appearance having a crystalline surface, cleaving with the greatest ease in three rectangular directions, giving flakes appearing grey by transmitted light but having no action on polarised light.

This selenide, of the formula $MnSe$, is isomorphous with the sulphide of manganese obtained under the same conditions by M. Mourlot.

Finally, I have been able to prepare fused selenide of manganese by the aid of a very beautiful method which consists of removing the oxygen from the seleniate of manganese by means of aluminium.

Powdered aluminium is intimately mixed with dried seleniate of manganese in the proportions indicated by the formula $3SeO_4Mn + 4Al_2 = 3SeMn + 4Al_2O_3$. The powder thus obtained is placed in a crucible and ignited by means of a piece of burning magnesium wire without the necessity of heating it.

The reaction is extremely violent: a long jet of flame escapes from the crucible, and, if it is then covered with a lid held firmly on, the melted mass causes this lid to adhere to the edge of the crucible, which, under the influence of the intense heat given off, becomes almost white-hot.

After cooling the melted selenide of manganese is detached from the cover and the sides of the crucible; it is nearly pure, and only contaminated on the surface with a little alumina.

If we do not take care to hold the cover on firmly when once the reaction is commenced, the whole mass will be thrown out of the crucible.

Properties.—Crystallised selenide of manganese, prepared in the electric furnace, has a density of 5.59 at 15°. It is easily attacked, even by dilute acids; fuming nitric acid transforms it into selenite of manganese.

Gaseous hydrochloric acid easily caused the evolution of SeH_2 .

When reduced to powder and treated with boiling peroxide of hydrogen containing a little hydrochloric acid, it is transformed into selenite of manganese.

It burns in chlorine, giving $MnCl_2$ and $SeCl_2$.

Oxyselenide of Manganese.—When we attempt to reduce seleniate of manganese by hydrogen at a high temperature, we obtain a green powder mixed with black selenide of manganese; at the same time selenious anhydride is given off, and condenses in the cooler parts of the tube.

This green powder, roughly separated, dissolves in hydrochloric acid, giving off SeH_2 ; it burns in oxygen, giving SeO_2 and saline oxide of manganese. It is an oxyselenide, analogous to the green oxysulphide of manganese which is formed on reducing sulphate of manganese by hydrogen.—*Bull. Soc. Chim.*, Ser. 3, xliii., No. 12.

LABORATORY NOTES.

By J. M. CAMP.

If apologies are required for the following notes, mine lie in the fact that they are detailed descriptions of some methods of analysis, partly new, that have stood the test of time and practical operations. They include a method for determining phosphorus in coke and coal, the determination of phosphorus in ores, pig-iron, and steel containing arsenic, and the determination of alumina as phosphate in ores and blast-furnace cinder.

PHOSPHORUS IN COKE AND COAL.

One of the requirements at the laboratory of which the writer has charge is the determination of the ash, sulphur, and phosphorus each day in an average sample of coke from all furnaces of the previous day's consumption. The fusion of the ash, consisting as it does of about 50 per cent silica, 30 per cent alumina, and 10 per cent sesquioxide of iron, with other bases, and its final evaporation to dryness to separate silica, is at best a tedious process which can not be safely hastened. The result is that the time of analysis is unduly prolonged; consequently the following scheme was evolved, yielding excellent results in the minimum of time.

The sample of coke partly dried, and ground to pass through a forty-mesh sieve, is delivered to the laboratory by the sampler on the afternoon of the day on which it is taken. This is dried at 100° C. for one hour, and when cool 5 grms. are weighed off into a 1½ inch porcelain crucible. This is left in the muffle furnace over night, and in the morning the lump of ash and any particles adhering to the crucible are transferred to a 30 c.c. platinum crucible, mounted on a platinum tripod. About 5 c.c. of dilute hydrochloric acid are now added, one acid to two of water, and about 10 c.c. of hydrofluoric acid, and the crucible and tripod are placed directly on the top of the chimney of an Argand burner, and the flame so regulated that the solution will not boil. In from 20 to 30 minutes

the solution is evaporated to dryness and dried to drive off the last traces of hydrofluoric acid, but not baked, as this will render some of the bases insoluble in the dilute acid to be subsequently used. When cool, about 15 c.c. of the same dilute hydrochloric acid are added, and the crucible warmed until all is in solution. The contents of the crucible are now transferred to a 12 c.m. evaporating dish, and 5 c.c. of strong nitric acid added. The bulk of the solution now aggregates about 75 c.c., and the solution is boiled for one or two minutes and then filtered, to remove any possible traces of silica or unconsumed carbon, into a sixteen-ounce flask.

The treatment now is the same as that previously described before this Society (*Proc. E. S. W. Pa.*, vol. xi., p. 251), to wit, the addition of 25 c.c. of strong ammonia, and then strong nitric acid until the precipitated iron and alumina are just dissolved, and then 5 c.c. in excess, making a total of about 25 c.c. strong nitric acid.

The solution is brought to 85° C. and 75 c.c. of molybdate solution, blown in by aid of a pipette, after which the solution is kept agitated for about five minutes, and is then filtered through a weighed filter-paper that has been dried at 115° to 130° C., and weighed between watch-glasses. The precipitate is washed with a 2 per cent solution of strong nitric acid, dried one hour at the above temperature, and weighed between watch-glasses; 1.63 per cent of its weight is taken for phosphorus.

In the case of coals the treatment is exactly similar, except that the coal is usually coked in a large platinum crucible, and then, to save the platinum crucible from the protracted heating, the coke is transferred to a porcelain crucible for complete combustion in the muffle preferably over night.

THE DETERMINATION OF PHOSPHORUS IN ORES, PIG-IRON, AND STEEL CONTAINING ARSENIC.

For the exact determination of phosphorus in ores, particularly manganese ores, coming as they do from all quarters of the globe, and in pig-iron or steel containing arsenic, although this impurity is not an every day occurrence, a method is very desirable wherein at some stage of the analysis the arsenic can be eliminated without a marked change in the essential details of the regular phosphorus determination, and without prolonging the time of the analysis beyond that of the regular routine work. Numerous experiments have been made by the writer along these lines, mainly in the attempt to reduce the concentrated ferric chloride solution to the ferrous state, and then volatilising the resulting arsenious chloride, but without marked success, until, acting on the suggestion contained in a paper by Mr. E. D. Campbell (*Journ. Amer. Chem. Soc.*, vol. vii.), describing briefly some experiments made by his students who used oxalic acid as the reducing agent, the following scheme was worked out. Excellent results were obtained up to 1 per cent arsenic. The method for ores only will be given; its application to pig-iron or steel will be readily seen.

Five grms. of the ground and dried sample are weighed off into a 12 c.m. porcelain dish with watch-glass cover, and 50 c.c. of strong hydrochloric acid added, and the solution boiled gently for about thirty minutes. It is now diluted with sufficient cold water to prevent cutting the filter-paper, and filtered into another dish of the same size.

This solution will contain all the unvolatilised arsenic in the ore, and it is placed on the steam-bath to go to dryness over night. The residue is burned and fused with the mixed carbonates, and the fusion allowed to harden around a platinum rod. The crucible is now warmed, and the greater bulk of the fusion removed on the platinum rod. This, while still hot, is placed in the dish with cover, in which the ore was originally dissolved, and containing 10 or 15 c.c. of water. Dilute hydrochloric acid is added to the crucible and warmed, and this process is repeated until all of its contents are removed and added to the dish containing the fusion. An excess of strong

hydrochloric acid is now added to dissolve any of the remaining fusion, and this dish is placed with the other on the sand-bath. In the morning, to the dish containing the dried mass from the original filtrate, 2 grms. of pure oxalic acid are added, and 50 c.c. of strong hydrochloric acid, and the solution, with watch-glass cover, evaporated to dryness by hard boiling, but not baked. When cool add 30 c.c. strong hydrochloric acid, and evaporate to first appearance of insoluble ferric chloride, remove from the light and add 10 c.c. strong nitric acid when the violent action has ceased, warm until all is in solution, dilute with cold water and filter into a sixteen-ounce flask, using 2 per cent nitric acid for washing. In the meantime, to the dish containing the fusion, dilute hydrochloric acid is added just sufficient to moisten, and enough hot water to dissolve the chlorides. This is warmed until all is in solution but the separated silica, and filtered into the same flask with the original filtrate. The phosphorus is now precipitated as above described for phosphorus in coke.

THE DETERMINATION OF ALUMINA AS PHOSPHATE IN ORE AND BLAST-FURNACE CINDER.

The greatest advantages which precipitating and weighing alumina as phosphate has over the hydrate precipitation is in ore analysis, where, with the latter method, the iron and alumina are weighed together, the iron being finally determined by solution of the precipitate and titration, or preferably titration in another portion of the same sample. All the errors of the entire manipulation are thus thrown on the alumina, the lesser dog. In the phosphate precipitation, however, the alumina is determined in a separate portion, and is responsible only for its own manipulative errors. In blast-furnace cinders, to determine the alumina, which is the laboratories' daily task, the determination of iron is entirely obviated. Most furnace managers are content to receive the silica and alumina in each day's cinders, counting the balance as bases, with a full analysis at regular stated intervals. The iron not being an essential component of the cinder, its determination in a cinder of a normal working furnace, where it is little more than a trace, is useless, while, with an abnormal working furnace, where it may be high, the manager has something else on hand to worry him of much more importance.

The filtration and washing of the phosphate precipitate are also much faster than the same operation with the hydrate precipitate. This statement applies particularly to cinders, where the large amount of alumina apparently coagulates the separated sulphur and precipitate settles rapidly, leaving the supernatant liquid practically clear and readily decanted. In ores, where there is much less alumina, more free sulphur is in suspension, and a partial but not aggravating clogging of the filter occurs.

One disadvantage of the phosphate method is that there is no end point to the washing of the precipitate, it being slightly soluble in the wash water, and after the tenth washing, showing with molybdate solution a fairly uniform volume of phosphomolybdate precipitate in equal volumes of the successive filtrates up to the twentieth washing. The addition of acetic acid and ammonium acetate to the wash water gave a slightly greater weight of aluminum phosphate from a slag of known composition, but still showed the phosphate in the filtrates up to the twentieth washing. It is important that the precipitate be washed thoroughly, otherwise the platinum ware, if used, will suffer.

The method as used on ore and cinders is as follows:—

To the cold hydrochloric acid filtrate from the silica of 1 grm. of ore or cinder diluted to about 400 c.c. in a No. 5 beaker, add 30 c.c. of a 10 per cent solution of ammonium phosphate and then ammonia until a faint permanent precipitate is formed; 1.5 c.c. of strong hydrochloric acid is now added, and for ores, on account of the greater bulk of iron present, 50 c.c., and for cinders 30 c.c. of a 20 per cent solution of sodium hyposulphite. The

beaker is now placed over the light and heated just to boiling. Now in the same graduate measure off 8 c.c. of strong acetic acid and 15 c.c. of a 20 per cent solution of ammonium acetate, and add to the boiling solution and boil ten minutes. If the latter is added before the solution is boiling, or near the boiling-point, the precipitate will be flocculent and difficult to filter. Remove beaker from the light and allow precipitate to subside, decant clear solution and wash precipitate on to the filter, and then wash ten times with hot water. Transfer precipitate to platinum crucible without lid and place in front part of the muffle; when paper is charred transfer crucible to hottest part of muffle till burned. Cool and weigh; 41.85 per cent of the weight is alumina.—*Proceedings of Engineers' Society of Western Pennsylvania*, xvi., No. 2.

NOTICES OF BOOKS.

A History of Chemistry from the Earliest Times to the Present Day. By ERNST VON MEYER, Ph.D. Translated, with the Author's sanction, by GEORGE MCGOWAN, Ph.D. Second English Edition, translated from the Second German Edition, with numerous Additions and Alterations. London: Macmillan and Co., Ltd. New York: The Macmillan Company. 1898. Pp. 631.

NOWHERE in antiquity do we find any endeavour to gain an insight into chemical processes by means of definitely planned experiments; though for hundreds, perhaps thousands, of years, chemical reactions have been used to attain certain ends, it was not until the first half of the sixteenth century, almost contemporaneously with the Reformation, that chemistry, losing its alchemistic tendencies, began to develop in a new direction.

In this "History of Chemistry" the author describes the development of chemical knowledge, and especially of the general doctrines of chemistry which have been gradually evolved from their earliest known beginnings up to the present day. In the general descriptions great emphasis has been laid upon the genesis of new ideas, and their gradual expansion into important dogmas or comprehensive theories.

In the special sections fundamental facts have been collected together, and these have been sifted and relegated to their proper branch of the science, thus forming as clear a description as possible of the state of chemical knowledge at any particular time.

The period of phlogistic chemistry was really an important one in the growth of the science; in spite of the false hypothesis which lay at the root of the theory, it was this theory, together with the work which resulted from it, that formed the foundation for the correct standpoints and researches of the succeeding period.

The latest period of chemistry, to which the present generation belongs, is closely associated with the name of Lavoisier, who turned the chemical science of his day into new paths; he demonstrated the supreme importance of the proportions by weight in chemical reactions: this applied more especially to the phenomenon of combustion and similar processes which Lavoisier was the first to explain correctly.

The next important step was the elaboration of the atomic theory founded by Dalton, which has gradually led up to Newland's and Mendeleeff's periodic system of the elements, Crookes's hypothesis of a primary material (protyle), and van 't Hoff's conceptions of the transformation and conservation of energy which have now come into general application in chemistry, more especially in the explanation of affinity-phenomena.

The enormous strides made in the science of chemistry in modern times is undoubtedly due to the greater facilities which have been available during the last few decades; the efforts made by Davy resulted in an increasing demand

for lectures with appropriate experiments. Practical instruction in chemical laboratories, as commonly carried out at the present day, was developed by Liebig; it was he who laid down the order in which the study of the various branches of the subject should succeed each other, and, thanks to the stimulus he exerted, a school was founded in his laboratory whose beneficial influence is felt at the present day all over the world.

The Carbonic Anhydride of the Atmosphere. By Prof. E. A. LETTS, D.Sc., Ph.D., and R. F. BLAKE, F.I.C., F.C.S. From the Scientific Proceedings of the Royal Dublin Society. Dublin: The University Press. 1900.

THE papers included in this number of the *Proceedings* of the Royal Dublin Society were read respectively on the 22nd June, 1898, and in April, 1899. The first paper deals with the methods of determining the amount of CO₂ in the air, with the authors' experiments on Pettenkofer's process as modified by them, and their experiments on the action of baryta water on glass and on silica, and the disturbing effect of soluble silicates on the delicacy of the phenolphthalein colour reaction.

Atmospheric carbonic anhydride, first shown to be present in the air by Dr. Black, of Edinburgh, probably between the dates 1752 and 1754, plays such an important part in nature, that the question of its amount, whether in the past, present, or future, is one of very great interest, not only to the chemist, but to the biologist, geologist, meteorologist, and student of nature generally.

The reciprocal action of plants and animals on air provides each with an important requisite, viz., carbon and oxygen respectively, and keeps in circulation the supply of carbon which is essential to the existence of both. If from any cause the amount of atmospheric carbonic anhydride should be largely increased, the existence of animals would be threatened, while if the reverse occurred vegetation would suffer, and animals would also stand in danger of eventual annihilation from the want of food, which they derive from the synthetic processes of the vegetable world.

This aspect of the question is considered in Part II., in which the causes of the variation of amount are studied, as well as the influence of day and night, vegetation, atmospheric precipitates, such as snow, fog, rain and mist, wind, the seasons, &c. Soil possesses the power of diminishing atmospheric carbonic anhydride at times, and then the air on the ground level contains less than that at higher levels; rain causes this, especially in the spring, that being the period when the soil is at its wettest. The volume, which will be read with great interest, concludes with a long and valuable list of the bibliography of the subject.

Elementary Practical Chemistry and Quantitative Analysis.

By FRANK CLOWES, D.Sc., Lond., &c., and J. BERNARD COLEMAN, A.R.C.Sc. Third edition. London: J. and A. Churchill. 1900. Pp. 282.

THE course of instruction given in this book is adapted to those students who do not need so thorough a chemical training as that which must be undertaken by one who has ambitions of becoming a professional and analytical chemist. The book is divided into two parts.

Part I. comprises a course of instruction on general chemistry, which gradually presents the principles of chemistry to the student by means of a selected series of experiments.

Part II. describes the analytical reactions of the commonly occurring elements and inorganic acid-radicles, and the methods of detecting them when they occur singly or in mixtures. At the end we find the usual tables for qualitative analysis, in which the elements are divided into the well-known groups and sub groups. Taken as a whole the book is sound, though not going very deeply into the subject.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 23, June 5, 1900.

Method of Decomposing several Metallic Perchlorides.—Echener de Coninck.—The author finds that various perchlorides are decomposed by the action of animal charcoal. He experiments on gold and platinum tetrachlorides, also on iron perchloride, in dilute aqueous solution. Other chlorides, such as NiCl_2 , CoCl_2 , FeCl_2 , MnCl_2 , ZnCl_2 , CuCl_2 , and MgCl_2 , undergo no change. The author proposes to continue this research on the decomposition of iron perchloride.

Conditions of Stability of Rotatory Power.—J. A. Le Bel.—If, instead of operating with bodies at ordinary temperatures, a temperature of about 100° is employed, it is seen that four different radicles united to the carbon will be necessary, but not more than sufficient to cause the rotatory power to appear in sodium amylic alcohol and in bromo-succinic acid; this is because these bodies present, under such conditions, a remarkable instability, in the same manner as active bodies containing nitrogen. On the other hand, when no new atoms are introduced from a new species, the stability is always increased by increasing the volume of the radicles.

Dihydroxylates.—M. de Forcrand.—The author examines the action of dilute peroxides of hydrogen in various proportions of dissolved alkaline bases at a temperature of about 15° . Numbers are given for the bases soda, potash, lithia, ammonia, and monomethylamine, CH_3N .

Hydrogenation of Acetylene in presence of Copper.—Paul Sabatier and J. B. Senderens.—A mixture of acetylene and excess of hydrogen is passed over copper recently reduced with hydrogen and cooled in this gas. In the cold no permanent action takes place. Combination starts at about 130° ; a considerable amount of heat being evolved. The reaction taking place is probably represented by the equation—

$93\text{H}_2 + 26\text{C}_2\text{H}_2 = 47\text{H}_2 + 17\text{C}_2\text{H}_6 + 11\text{C}_2\text{H}_4 + 27\text{C}_2\text{H}_4$
By increasing the proportion of acetylene, the special action of copper on acetylene begins to be noticed, cuprene being formed.

Cuprous and Mercurous Organo-metallic Compounds of Diphenylcarbazone.—P. Cazeneuve.—In a preceding paper the author noticed the metallic derivatives of diphenylcarbazone corresponding to the general formula $\text{CO} \begin{smallmatrix} \text{NM}'\text{—NH—C}_6\text{H}_5 \\ \text{N} = \text{N—C}_6\text{H}_5 \end{smallmatrix}$. Under certain experimental conditions, the cuprous and mercurous derivatives

corresponding to the formulæ $\text{CO} \begin{smallmatrix} \text{NCu—NCu—C}_6\text{H}_5 \\ \text{N} = \text{N—C}_6\text{H}_5 \end{smallmatrix}$

and $\text{CO} \begin{smallmatrix} \text{NHg—NHg—C}_6\text{H}_5 \\ \text{N} = \text{N—C}_6\text{H}_5 \end{smallmatrix}$ can be obtained. These substances easily lose the metal, being transformed into carbodiazide, $\text{CO} \begin{smallmatrix} \text{N=N—C}_6\text{H}_5 \\ \text{N=N—C}_6\text{H}_5 \end{smallmatrix}$, a substance which has not yet been isolated, E. Fischer having only prepared the sulphocarbodiazine, $\text{CS} \begin{smallmatrix} \text{N=N—C}_6\text{H}_5 \\ \text{N=N—C}_6\text{H}_5 \end{smallmatrix}$.

Acidimetry.—A. Astruc.—The author continues his researches on this subject, considering now the behaviour of various organic acids with regard to certain indicators. The special acids considered are isethionic, sulphanilic, meconic, and mellic acid.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxxiii., No. 4.

Thermic Examination of Normal Adipic Acid.—G. Massol.—The author has determined the heats of neutralisation and formation of the potash salts of normal adipic acid. This acid, when prepared and purified carefully, melts at 146.5° . It is anhydrous, and the acidimetric assay with phenolphthalein gave 100.2 per cent. The acid is too slightly soluble in water (1 per cent at 15°) for its heat of solution to be determined. The solution of 1 molecule of adipic acid in a solution of potash (1 mol. in 4 litres) is easily effected at the ordinary temperature, and produces a considerable quantity of heat:—

$\text{C}_6\text{H}_{10}\text{O}_4 \text{ sol.} + \text{KOH diss.} = \text{C}_6\text{H}_9\text{O}_4\text{K diss.} \dots \dots + 6.93 \text{ cal.}$

This figure represents the heat of neutralisation of the acid salt, less the heat of solution of the solid acid. The addition of a second molecule of potash to the acid salt gives the neutral salt:—

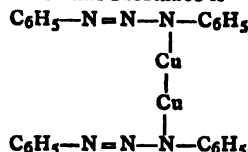
$\text{C}_6\text{H}_9\text{O}_4\text{K diss.} + \text{KOH diss.} = \text{C}_6\text{H}_8\text{O}_4\text{K}_2 \text{ diss.} \dots \dots + 13.41 \text{ cal.}$

The total heat of neutralisation representing the formation of the neutral salt is $+20.34$ cal. The solution of adipate of potash, neutral to phthalein and litmus, gave the neutral anhydrous salt by evaporation and desiccation at 100° . This salt dissolves in water with the evolution of heat: 1 mol. (= 222 grms. in 1 litre) gives off $+2.67$ cal. The heat of formation of the solid neutral adipate, calculated from the above data, is as follows:—

$\text{C}_6\text{H}_{10}\text{O}_4 \text{ sol.} + 2\text{KOH sol.} = \text{C}_6\text{H}_8\text{O}_4\text{K}_2 \text{ sol.} + 2\text{H}_2\text{O sol.} \dots \dots + 45.45 \text{ cal.}$

This high figure shows that adipic acid is an energetic acid quite comparable with its homologues, succinic, glutaric, and suberic acids, which, under the same conditions, give off $+46.50$, $+44.23$, and $+44.76$ cal.

A Cuprous Salt of Diazoamidobenzene.—L. Meunier and A. Rigot.—To a very concentrated solution of diazoamidobenzene in alcohol, freshly prepared finely-divided copper is added, and the mass well stirred for twelve hours. In this manner a yellowish-brown precipitate is obtained, which is washed with alcohol to eliminate the uncombined diazoamide. The residue which contains the excess of copper is exhausted with boiling benzene, which dissolves the organo-metallic substance. When evaporated, the benzenic solution leaves a residue formed of needles, which, after being purified by several crystallisations in boiling benzene, take the form of small yellowish-orange felted crystals, which may be dried at 50° . The formula of this substance is—



This cuprous salt is insoluble in water, alcohol, ether, and ligroin; it is slightly soluble in benzene, especially when heated; it decomposes at about 270° . Ordinary nitric acid decomposes it with explosion and incandescence. When a current of hydrochloric acid gas is passed through a benzenic solution of the cuprous compound, a yellowish-white precipitate is formed immediately, and the solution becomes colourless; this precipitate, collected and washed with ether, forms a bright yellow amorphous powder, soluble in water, in which solution sulphuretted hydrogen gives a precipitate of sulphide of copper, and nitrate of silver a precipitate of chloride of silver.

Some Amines containing the Camphor Radicle.—G. Blanc.—Already noticed in this column.

Rapid Method for the Estimation of Clay in Soils.—F. Poquillon.—Already inserted in full.

MISCELLANEOUS.

Estimation of Sulphur in Naphtha.—A. Lidof.—The author dissolves 1 grm. of naphtha in pure ether, and mixes it in a mortar with 30 grms. of a mixture of powdered NO_3K and Na_2CO_3 (17 parts of NO_3K and 13 parts of Na_2CO_3). After evaporating off the ether he throws the residue, a little at a time, into a large platinum crucible (250 to 300 c.c.) heated to redness. He then estimates the SO_4H_2 with BaCl_2 in the usual manner. Although a large part of the sulphur contained in the naphtha is in the form of volatile organic compounds, the method gives sufficiently exact results for ordinary practical purposes.—*Journ. Soc. Phys. Chim. R.*, vol. xxxi., p. 567.

Methods of Preparing Graphitic Acid.—L. Staudenmaier.—The author gives two methods for the preparation of this body in small quantities as a lecture experiment. Then follows a process for the preparation of this acid on the large scale. For this purpose a well powdered good quality graphite, such as commercial Ceylon graphite, is preferred; 250 grms. of this substance are treated with a mixture of 7 litres of sulphuric acid and 3 litres of nitric acid $\text{D} = 1.37$. After adding about 9 kilograms of powdered chlorate of potassium, the whole is left for six days at a low temperature; the supernatant liquid is then decanted, and the greenish product obtained is washed with warm water, then with nitric acid $\text{D} = 1.4$, diluted with one volume of water. The oxidation is finished by means of permanganate of potash and hot sulphuric acid, and the permanganic acid formed is decomposed by heating the mixture. The operation is terminated by the addition of peroxide of hydrogen to get all the manganese into solution, and washing the graphitic acid formed.—*Berichte*, vol. xxxi., p. 1394.

New Method for the Estimation of Glycogen.—Pflüger and Nerking.—If we dissolve meat by means of boiling caustic soda, we can separate, in the liquor obtained, the glycogen from the proteic substances by the following method:—To 100 c.c. of liquor containing 3 grms. of caustic soda or potash we add 10 grms. of iodide of potassium and 50 c.c. of alcohol at 96 per cent; the glycogen is entirely precipitated. If we separate the precipitate by filtration, and wash the precipitate on the filter with a solution prepared by mixing 50 c.c. of alcohol at 96 per cent with 100 c.c. of water containing 3 grms. of caustic potash and 10 grms. of iodide of potassium, and if, finally, we re-dissolve this washed precipitate in water, we find that this solution which contains all the glycogen of the original liquid does not contain any proteic materials. We thus have at our disposal a more simple and as exact a process as that of Brücke for separating the glycogen from proteic matters, a process which can be used with great advantage for the preparation and estimation of glycogen in extracts of tissues.—*Pflüger's Archiv*, vol. lxxvi., p. 531.

The Solubility of Copper in an Alkaline Solution of Gelatin.—A. Lidof.—The author has observed that the violet solution obtained by the reaction of salts of copper on an alkaline solution of gelatin is decomposed when heated under pressure in a Lintner apparatus, and that metallic copper is deposited. The same result is obtained in the cold ten or fifteen hours after formic aldehyde has been added. The precipitate, however, is not pure copper, but contains a certain amount of organic matter, and only 92 per cent of copper. From this the author is inclined to the opinion that the so-called biuret action is nothing else than the passage of copper into solution; that is to say that the salts of copper are reduced by the organic matter, and that the metal takes a soluble colloidal form. Acting on this opinion the author has made the following experiment:—A spiral of copper weighing 39.487 grms., quite free from oxide, was plunged

into a solution of 150 grms. of water, 13.6 grms. of gelatin, and 14 grms. of KOH . On the following day the liquid already had a violet tint, which, as time passed, became of a deeper hue. After forty-eight days the spiral had lost 1.301 grm., or 3.54 per cent of its weight.—*Journ. Soc. Phys. Chim. R.*, vol. xxxi., p. 571.

The Action of Heat, Dilute Acids, and Alcohol on Albumin.—A. Panormof.—If we dialyse albumin with solutions of 0.05 per cent to 0.5 per cent of HCl , HBr , PO_4H_3 , $\text{P}_2\text{O}_7\text{H}_4$, and PO_3H , soluble compounds with these acids are formed, except with the last. The rotatory power of the albumin has increased, especially with the HCl , and it increases still more when heated to 100° ; but analysis always shows the same composition for albumin combined with acids. The variation of the rotatory power appears to be due to a polymerisation of the albumin. If we decompose the compound formed with the acid without heating it, we recover the original crystallisable albumin; if we heat it to 100° we get an amorphous body with different properties, but having the same composition. In any case the albumin has the empirical composition $\text{Alb} = \text{C}_{235}\text{H}_{422}\text{N}_{63}\text{O}_{85}\text{S}_3$. The compounds obtained by dialysis by the author and by M. Worms, are:—

	Per cent.
$\text{Alb}(\text{HCl})_5$ with HCl at	0.1
$\text{Alb}(\text{HBr})_3$ with HBr at	0.1
$\text{Alb}(\text{PO}_4\text{H}_3)_2$ with PO_4H_3 at	0.05
$\text{Alb}(\text{PO}_4\text{H}_3)_3$ with PO_4H_3 at	0.2
$\text{Alb}(\text{PO}_4\text{H}_3)_4$ with PO_4H_3 at	0.5
$\text{Alb}(\text{P}_2\text{O}_7\text{H}_4)_3$ with $\text{P}_2\text{O}_7\text{H}_4$ at	0.2
$2\text{Alb}(\text{P}_2\text{O}_7\text{H}_4)_7$ with $\text{P}_2\text{O}_7\text{H}_4$ at	0.5

If we heat $\text{Alb}(\text{P}_2\text{O}_7\text{H}_4)_3$ or $2\text{Alb}(\text{P}_2\text{O}_7\text{H}_4)_7$ with the acid solutions which have furnished these compounds we obtain $\text{Alb}(\text{PO}_4\text{H}_3)_3$ or $\text{Alb}(\text{PO}_4\text{H}_3)_5$. Albumin, dialysed with water and then filtered, does not lose its solubility by concentration in vacuo; it, however, ceases to be soluble when heated in sealed tubes, or when precipitated by a mixture of alcohol and ether. In these latter cases a polymerisation is probably produced. According to the author acids, heat, and alcohol easily polymerise albumin; it is this that proves the constancy of the composition and the variation of certain properties, such as the rotatory power and the solubility.—*Journ. Soc. Phys. Chim. R.*, vol. xxxi., p. 556.

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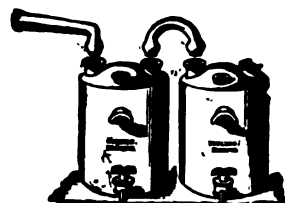
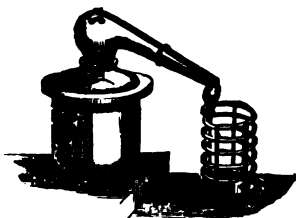
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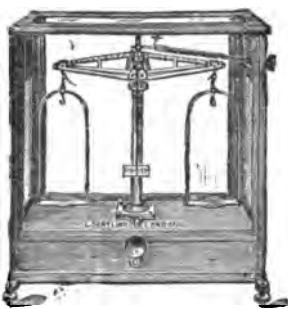
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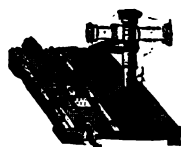
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VOL. LXXXII., No. 2120

THE SENSITIVENESS OF SILVER AND OF SOME OTHER METALS TO LIGHT.*

By Major-General J. WATERHOUSE, I.S.C.,
(late Assistant Surveyor-General of India).

(Concluded from p. 4).

Images Formed on Both Sides of Exposed Glass Plate.—In connection with this action through glass, a curious result obtained on a plain glass plate may be mentioned. A piece of clean glass plate was exposed for a day and a half in October under the same aluminium screen, with another glass plate over it, so as to protect the surface from the air. It showed a clear breath image, but mercury vapour did not produce any image. Iron development, however, brought out part of the design very clearly, the silver depositing upon the unexposed parts, and giving an image darker and clearer than the ground. The image of the design appeared by breathing on both sides of the exposed glass plate, so that the action of light went through both plates.

Effect Produced on Varnished Silver Glass.—With the same object of ascertaining whether the images could be produced when the silver surface was protected from the air by varnish, a silvered glass plate was coated over one half with photographic negative varnish, and then exposed in the usual way, under the cut-out paper mask and mica screen, in October. After exposure for two days the image of the black paper mask, the cut-out initials, and edges of the mica screen were not reproduced so clearly as usual, though they appeared readily enough on the unvarnished half when breathed upon. The distinct heightening of the effect under the varnished part is apparently due to some chemical combination, probably oxidation, or the formation of an organic silver compound sensitive to light. When the varnish is removed the strong image remains, and there is a distinct change of colour in the exposed part of the silver surface, which takes a sort of yellowish olive grey tint. Recent repetitions of the experiment with silvered glass, and with a plate of nearly pure silver, gave exactly the same results. It may be noted that with iodised silver plates the same varnish has been found to exercise a decidedly retarding effect on printed-out images.

Probable Cause of the Effects Described.—As regards the cause of the peculiar effects described, and the nature of the visible and invisible but developable images produced by the action of light upon plain silver surfaces, it is very difficult to give any definite opinion. It would seem that in this, as in most photographic processes, the first action of light is principally molecular, but if the exposure be prolonged and takes place in the air under ordinary conditions, there is a certain chemical decomposition of the surface of the plate, and the impressed image becomes distinctly visible.

Moser.—Moser was of opinion that the action of light does not necessarily consist in the separation of two chemically combined bodies, even in the Daguerreotype, in which process he maintained there is no separation of iodine from silver under the action of light. (We now know that the iodine evolved is absorbed by the underlying silver). He brought forward his experiments on pure surfaces of silver, where, he says, there can be no possibility of a chemical action to show that the effects of the Daguerreotype could be produced in quite a different way.

Waidele.—Waidele (*Pogg. Ann.*, 1843, vol. lix.), although he does not refer to Moser's observation of the direct action of light upon silver, and concerns himself more with the contact actions attributed to invisible light, concludes that Moser's effects are not produced by any action of invisible light, but must be rather explained by the action of atmospheres on bodies and the absorption of gases. He points out that polishing powders, such as tripoli, charcoal, &c., ordinarily contain moisture and absorbed gases, for which they have a strong attraction, and if used in this state they give up these vapours to the surface polished. If, however, they are heated to a red heat to drive off all moisture and gases, they then act as absorbents, drawing out the moisture and gaseous impurities from the surface of the metal, and producing a far purer and more perfect polish. He also notes the effect of carbonic acid, hydrogen, and other vapours on iodised silver Daguerreotype plates.

So far as my experiments go, I do not think that the nature of the polishing materials has had much influence on the results obtained, though it is an interesting point which might be looked into.

Roscoe.—Roscoe ("Watts's Dict. of Chem.," 1872, ii., p. 805) also attributes the Moser effects to absorbed gases, and says:—"Almost all of the singular phenomena first investigated by Moser, and ascribed by him to the action of latent light, may be more rationally explained by the authenticated facts of the absorption of gases by solid bodies." He also refers more particularly to the contact or "breath" images produced by laying coins, &c., upon polished plates of metal.

Action of Gases.—It is evident that if there be any chemical action on the silver under the influence of light, it must be produced by the agency of gases or vapours contained:—

- (i.). In the silver itself.
- (ii.). In the layer of air or condensed gases in immediate contact with the plate.
- (iii.). In the surrounding atmosphere.
- (iv.). In the masks or coverings under which the exposures are made.

What the exact nature of the decomposition may be, and to which of these agencies it is attributable, is not easy to ascertain, nor does the appearance of the visible image give much clue to it. From the dull-grey colour of the exposed parts it seems, however, probable that oxygen plays an important part in the action; but whether the oxygen is drawn from the air or is disengaged from the metal itself, or whether both actions take place with interactions of other gases occluded in the metal itself or present in the atmosphere in contact with its surface, there is nothing so far to definitely show. It seems also probable that, as in many other photographic processes, the presence of watery vapour is necessary to bring about the decomposition. These points require further investigation at a time of the year when the light is bright, and the effects can be observed under the most favourable conditions. From the fact that in most of my experiments the external air has been excluded by the outer glasses of the printing frames, we may, I think, conclude that the effects are due more to the gases or vapours occluded in or attached to the silver surface or to the screens, rather than to any outside atmospheric influences.

Oxygen Found in Silver by Graham.—With regard to the presence of oxygen in silver, Graham found that silver heated and allowed to cool in oxygen could occlude 0.745 volume of oxygen, which was permanently fixed in the metal at all temperatures below an incipient red heat. It did not tarnish the bright metallic surface of the silver, or produce any appearance suggestive of the oxidation of a metal. He further showed that silver in the form of sponge can occlude eight volumes of oxygen without any visible tarnish. Silver appears to have a relation to oxygen similar to that exhibited by platinum, palladium, and iron to hydrogen. Silver can also occlude variable quantities

* A Paper read before the Royal Society, May 31, 1900.

of hydrogen, nitrogen, and carbonic acid (*Phil. Trans.*, 1866, p. 434).

Dumas's Observations.—According to Dumas (*Comptes Rendus*, 1878, vol. lxxvii., p. 69), 1 kilo. of pure silver, prepared by fusion with borax and nitre, was heated in a vacuum to a temperature not exceeding a dull red, about 500° or 600° C. The evolution of gas continued for about six hours, and it was received over mercury. The gas given off was pure oxygen, amounting to 47 c.c. at 0°, and 760 m.m. of pressure for 1 kilo. of silver, and weighing 82 milligrams. In two other experiments, in which silver was fused in a more oxygenated atmosphere, as much as 158 c.c. of oxygen, weighing 226 milligrams, and 174 c.c., weighing 249 milligrams, were obtained from 1 kilo. of pure silver in each case. He found also that silver which contains oxygen does not lose it all in a cold vacuum.

Mallet's Observations.—Professor J. W. Mallet, in some investigations on the Atomic Weight of Aluminium, notes these results obtained by Dumas, and states that in his own observations of the same kind, but using a lime support for the silver heated in a hard glass tube to a moderate redness, he obtained only 34.63 c.c. of oxygen from 1 kilo. of silver (*Phil. Trans.*, 1880, p. 1020).

These different observations show that silver, apparently pure, may contain a very considerable quantity of oxygen, and I believe it is a well-known fact, especially in the Mints, that oxygen is nearly always present in silver, with or without hydrogen, carbonic acid, and other gaseous impurities in much smaller quantities.

Whether occluded oxygen is the cause of the photographic effects produced on surfaces of apparently pure silver might readily be proved by extracting all the oxygen from a silver plate by the above methods, and then exposing the resulting purified silver to light, a similar plate from which the oxygen had not been extracted being exposed at the same time. I have not the appliances at my disposal for making this experiment.

Effects of Heating Silver Plates to Redness.—With regard, however, to the effect of simply heating the silver plates to redness, the following experiments may be of interest, and seem to show that the heating, whether accompanied by subsequent "blanching" or "pickling" in dilute sulphuric acid or not, causes a distinct loss of sensitiveness, and in some cases may destroy it entirely.

A piece of thin, pure silver plate was first of all heated to a red heat over a spirit-lamp, then plunged into dilute sulphuric acid, and, after being washed with distilled water and dried, was exposed for about two days and a half, partly in sunshine, at the end of September last, under a screen of mica carrying a cut-out design in tinfoil, which had also been passed through the flame of the lamp. No visible image was produced, nor did one appear by breathing on the plate. Development with acid ferrous sulphate and silver nitrate brought out traces of the opaque tinfoil mask, and of parts cut out of the mica. The free ends of the plate, which were exposed to the air all the time, did not show any distinct attraction for the developer.

Another plate of pure silver, well cleaned with "meteoric" polish (No. 2) and exposed as usual under a mica screen with black paper mask, only one day longer than the last plate, gave a very strong visible image requiring no development. There must, therefore, have been a considerable difference in the conditions of the surfaces of the two plates.

In the plate which was heated and then treated with dilute sulphuric acid, it may be assumed that any surface layer of condensed gases must have been destroyed, and a surface of pure metal exposed—which, as one would expect, was not visibly sensitive to light. In the other case, in which the plate was probably oxidised (it had been lying by for several years), and was simply well cleaned with a dry polishing powder, the surface of the metal was left so sensitive to light as to give a strong visible image. This difference of action in two plates, which were ex-

posed to the same conditions of light, seems to show that the effects produced by light on surfaces of metallic silver are chemical rather than merely molecular in their nature.

A repetition of this observation gave similar results. Further, a plate of thin silver foil, well heated over the spirit-lamp and exposed under a mica screen, which was also heated, for two days, including some hours of sunshine, showed no image, even on development with mercury, although distinctly visible images had been obtained on the same piece of foil when cleaned in the ordinary way and exposed under the same or similar screens. Further experiment on this point is still necessary.

Effects on Silver Surfaces Fumed with Acid and other Vapours.—A good many experiments have been made with silvered glass plates or silver foil, fumed with various vapours, which might possibly be present in small quantities during the exposure to light, among them hydrogen peroxide, nitric acid, ammonia, also sulphurous acid; these plates have all shown visible and developable images of a somewhat similar character to those formed on the plain metal.

Nitric Acid.—By fuming silvered glass plates with ordinary pure nitric acid (1.420) or, better, with the same acid diluted with an equal volume of water, very clear positive printed-out images have been obtained, even with the comparatively short exposure of one hour in the February sun. With a longer exposure the image was clearer, the exposed parts appearing lighter than the protected parts, as is also the case with the plain silver surfaces.

Dilute Ammonia.—Dilute ammonia containing one part of the solution at 0.880 in 30 of water gave a similar image, but the film was not so sensitive as with the nitric acid. Further experiments have, however, to be made.

Dilute Sulphurous Acid.—Dilute sulphurous acid, formed by acidifying a solution of sodium sulphite with sulphuric acid, gave a weak image of much the same character.

Hydrogen Peroxide Solution.—Silvered glass plates, fumed with various strengths of hydrogen peroxide solution, have given fairly sensitive films, sometimes a little white in appearance, but as a rule the printed-out images produced on exposure to light are just the reverse of those produced on plain plates and those fumed, as noted above, *i.e.*, the parts exposed appear darker than the unexposed parts, and do not attract mercury vapour, so that a positive picture is produced from an ordinary black and white negative. By long exposure this effect is sometimes reversed. The visible images produced on silvered glass plates fumed with the peroxide quickly fade away, though the details may still remain visible on breathing. The same effect of fading out has also been observed on plain silver plates.

Rain or Boiled Distilled Water.—Silver plates exposed under fresh, clean rain water, or boiled distilled water, gave distinct images on development with mercury, but not very readily, and further observations are necessary.

Exposures in Fluid Hydrocarbons.—In order to ascertain whether the images would be produced on silver surfaces exposed in fluid hydrocarbons containing no oxygen, silvered glass and pure silver foil were exposed in fluid paraffin, benzole, xylol, and toluol.

Paraffin.—In clear, colourless, fluid paraffin very strong dark olive-yellow images were produced by light on silvered glass and silver foil on the parts exposed freely in the fluid or under the parts cut out of a black cardboard mask, but under a mica screen, which covered part of the black mask, and of the plate itself, there was no darkening action whatever. The darkening appears to be due to sulphuration, but why it should not occur under the mica is not clear, unless the latter cuts off the rays which are active in producing it.

Benzole.—Silver foil well cleaned and exposed in ordinary rectified benzole under a cut-out black paper mask, and also half covered with mica, showed very clear,

yellow images of the cut-out design on the part uncovered by the mica, apparently by decomposition of thiophene or other sulphur compound present in the benzole. This darkening required strong sunshine to produce it, and on prolonging the exposure in the sun the metal became quite bronzed in the uncovered exposed part, while the part protected by the mica also took a light tint. It may be noted that, as a rule, the backs of these slips of foil (which were exposed in test-tubes) were not protected by any covering, but showed no perceptible change of colour: the darkening of the exposed foil was therefore solely an effect of light.

Effect of Heating the Foil.—A piece of the same foil was exposed in the same way in the same benzole after being heated to redness. This did not show such a strong image, even by long exposure in the sun.

A piece of pure foil (assay foil) was exposed in a purer sample of benzole, and also gave a distinct yellow image as soon as the sun acted upon it after some days' exposure, the light being dull till near the end of the period. Another piece of the same foil, heated to redness and exposed at the same time, only showed a very pale yellowing.

Xylo.—A similar very faint, but browner, change of colour was noticed on a piece of the same foil, not heated, exposed for several days in commercial pure xylo.

With toluol, similar results were obtained after long exposure.

Effects of Light upon other Metals.—A few observations have been made as to the action of light upon other metals, but with the exception of lead none of them have proved very sensitive, and further work is necessary in good weather.

Gold.—Images developable with mercury have been obtained on gold leaf by prolonged exposures, but scarcely any trace of a developable image could be obtained on some highly-polished well-gilt buttons exposed for several days in good sunshine in October.

Lead Foil.—On pure lead foil I have obtained a very distinct darkened image visible after exposure. Very distinct darkening was also produced on lead foil exposed in pure benzole.

Copper.—On copper distinct visible images have been obtainable sometimes, but the metal is not so sensitive as silver to light, though I have readily obtained heat images on it developable by mercury. Pure copper foil exposed in sunshine in pure benzole showed a distinct darkening, but exposed in xylo it did not change colour.

Nickel, Platinum, Aluminium, Palladium.—Nickel, aluminium, and platinum appear to be quite insensitive to light, but with a small button of palladium, kindly lent to me by Mr. T. Bolas, which was exposed for some days under a black paper mask, there appeared a slight but fairly distinct trace of deposit of mercury on the exposed parts. The spot was too small to make sure of.

Trial with Röntgen Rays.—With the kind assistance of Mr. F. H. Glew I was able to make a few experiments with the Röntgen rays upon silvered glass and pure silver plates, exposed for several hours to the rays, but without any visible or developable effect.

The above is a summary of my observations in this direction so far as they have gone. I hoped to have made them more complete, but the dull winter weather has been very much against such work. I hope, however, to go on with it during the summer, but think it advisable to publish the results already obtained now, in order that others may be able to extend them at the same time. They show, I think, that most of the phenomena that occur by the exposure of ordinary photographic plates containing haloid compounds of silver can be observed upon a plain silver plate exposed to light in the air under ordinary conditions. It seems not impossible, therefore, that the key to the hitherto unsolved problem of the production and constitution of the so-called "latent photographic image" may be found within these limits.

THE EIGHTH GROUP OF THE PERIODIC SYSTEM AND SOME OF ITS PROBLEMS.*

By JAS. LEWIS HOWE,

In the early work of Newlands and of Mendeléeff, which subsequently developed into the periodic law, a serious difficulty was met with in dealing with iron, cobalt, nickel, and the metals of the platinum group. In Newlands' modified statement of his law of octaves he says: "The numbers of analogous elements, when not consecutive, differ by seven or by some multiple of seven." Thus we find him grouping (CHEM. NEWS, 1866, xiii., 130) cobalt and nickel under a single number; so rhodium and ruthenium; so also platinum and iridium. Cobalt, nickel, palladium, platinum, and iridium are considered by him analogous elements, each occupying the first place in the octave to which it belongs; iron, rhodium, ruthenium, and gold are analogous elements, each occupying the seventh place in its octave, while osmium is included with copper and silver as the second members of their octaves. There was here an easily recognised inconsistency which was not cleared up till many years later Seubert was led by the study of the periodic law to revise the atomic weights of these metals.

In his first summing up of the principles of the periodic law in 1869, Mendeléeff concludes that "elements which are similar, as regards their chemical properties, have atomic weights which are either of nearly the same value (e.g., platinum, iridium, osmium) or which increase regularly (e.g., potassium, rubidium, cesium)" (*Ann. Phys. Chem. Soc. Russ.*, 1869, i., 60). So in most schemes for representing the periodic system, each triplet of these elements is considered as a single element, and because even then they do not seem to fall into regular periodic arrangement they are cast out, Ishmael-like, into an anomalous eighth group. This is doubtless the reason they have been relatively so much neglected by chemists, and possibly it is not incorrect to say that the chemistry of these metals is less known than that of any other group of well characterised elements. Yet there are certainly no nine nearly-related elements which present so many interesting chemical problems, whose solutions will so much further our knowledge of chemistry in general. It is the purpose of this address to attract the attention of the members of this section to this group and some of its many problems.

The ordinary division of these nine metals is into three groups, viz., the common metals, iron, cobalt, and nickel, with an atomic weight of from 56 to 59, and a specific gravity of 7.8 to 8.9; the lighter platinum metals, ruthenium, rhodium, and palladium, with an atomic weight of 101.5 to 106.5, and a specific gravity of about 12; and the heavy platinum metals, osmium, iridium, and platinum, of atomic weight 191 to 195, and specific gravity 21.5 to 22.5.

Of these metals iron alone can be considered abundant, and was the only one known until the eighteenth century. The ores of cobalt and nickel have been known for over two centuries, but the probable presence of a new metal in cobalt ore was first pointed out by Brandt (*Akta Reg. Soc. Sci. Upsala*, 1735, 33) in 1735, and nineteen years later Cronstedt (*K. Svenska Vet. Akad. Handl.*, 1751, 293) determined the existence of nickel. Both of these discoveries were several decades after confirmed by Bergman (*Opusc.*, Diss 20, 1775; 1780, xxiv., 12 and 14; 1779, xxv., 31 and 33).

It is, however, a curious and interesting fact that a coin of Badria (*Pogg. Ann. der Phys.*, 1870, cxxxix., 507) of a date more than two centuries before Christ has been found containing 20 per cent of nickel, and hence quite similar in composition to our modern "nickels." There

* An Address delivered before the American Association for the Advancement of Science, Section C.

seem, however, to be no references in ancient literature which would indicate that attention was ever attracted by nickel or any of its compounds.

Turning to the platinum metals, the first recognised mention of platinum seems to be in the "Relacion Historica" of Don Antonio de Ulloa (vol. i., lib. vi., cap. 10, p. 606), published first in 1748, the book being an account of the French expedition of 1735 to the western coast of

From the mention here made, platinum is evidently a well-known substance, and it is by no means improbable that earlier definite mention of platinum may yet be found. In this connection it is interesting to note that there have been many efforts to show that platinum was known at much earlier periods. Scherer (*Allg. J. Chem.*, Scherer, 1801, vi., 633) considers, from a passage in Balbin's "History of Bohemia" (P. I., ch. xiv., p. 4), that platinum was

PERIODIC TABLE, BY F. P. VENABLE—MODIFIED.

H														He		
Li		Gl		B		C		N		O		F		Ne		
Na		Mg		Al		Si		P		S		Cl		Ar		
																
K	Cu	Ca	Zn	Sc	Ga	Ti	Ge	V	As	Cr	Se	Mn	Br	Fe	Co	Ni
														[82]	[84]	
Rb	Ag	Sr	Cd	Y	In	Zr	Sn	Cb	Sb	Mo	Te	—	I	Ru	Rh	Pd
Cs	†	Ba	†	La	†	Ce	†	•	†	•	†	•	†	•	•	•
•	Au	•	Hg	•	Tl	•	Pb	Ta	Bi	W	†	•	†	Os	Ir	Pt
•		•		•		Th		•		U						
† Series.	— Series.	+	—	+	—	+	—	+	—	+	—	+	—			

* Possible + series elements.

† Possible — series elements.

— Khamanganese.

South America to measure an arc of the meridian on the equator. The passage reads: In the district of Chocó (Columbia), which contains many placer mines, are also found some, the gold of which, occurring mixed with other substances as metals and rocks, or being enveloped in them, requires the use of quicksilver for its extraction, and sometimes ores are found which are not worked, because of the *platina* in them (a mineral of such resistance that it is not easy to break it, nor to crush it upon an anvil), for this substance is not affected by calcination, nor is there any means of extracting the metal which it contains, except at the cost of much labour and expense.

known to the Bohemian Jesuits toward the end of the seventeenth century, occurring in the Riesengebirge, for he speaks of a kind of gold so white that one would swear it was silver,* were it not for its weight, ductility, infusibility, and insolubility in nitric acid. Julius Scaliger's "Exercitationes Exotericæ de Subtilitate," published at Frankfort in 1601, makes mention of an infusible metal found in the mines of Mexico and Darien, which might

* Aurum album, quod argentum esse jurares, nisi auro familiares proprietates aliud suaderent, pondus scilicet, extensibilitas, vis cludendi ignem et aquam fortem, solubilitas in aqua regis, &c.

seem to indicate platinum.* There have been, moreover, a number of efforts to show that platinum was known to antiquity under the names of *electrum* or of *plumbum candidum*. Cortinovis (*Opuscoli Scelti Sulla Scienze, &c.*, Milano, 1760) considers that the *electrum* of the ancients was platinum, and proves it to his own satisfaction from the Bible, from classic historians of nature and art, from poetry and from Homer.† A similar view is held by Schweigger (*J. Prakt. Chem.*, 1845, xxxiv., 385), where Pansanius (v., Chap. 12, p. 406, Ed. Casaub.) is quoted as mentioning *electrum*, from which Augustus had made columns, as occurring in nature in the sands of the river Eridanus, and as being very rare, hence much more valuable than the other kind of *electrum* which is merely an alloy of silver and gold. In 1850 Paravey (*Comptes Rendus*, 1850, xxi., 179) makes a strong effort to show that Pliny, when speaking in his Natural History of *plumbum candidum*, refers to platinum. Pliny speaks, indeed, of a lead heavier and more ductile than gold, and in Book 34, Chapter 16, gives a definite description of it. To use the quaint translation of Philemon Holland, Doctor of Physick, published first in 1601:—

"Now insoeth the discourse of lead and the nature of it, of which there be two principall kinde, the blacke and the white. The richest of all, and that which carrieth the greatest price, is that which we in Latine name *plumbum candidum*, i.e., the white bright lead, and the Greeks *Cassiteron*. But I hold it a meere fable and vaine tale, that all of it is fetched as farre as from the Islands of the Atlantick Sea, and that the inhabitants of those parts doe convey it in little twiggen boats, couered all ouer with feathers. For the truth is that there is found of it in these daies within Portugall and Gallacia, growing ebbe upon the opmost face of the earth, being among the sands, of a black colour, and by the weight only is knowne from the rest of the soile: and here and there among, a man shall meet with small stones of the same stuffe, most of all within the brookes, that be dry sometimes of the yere. This sandie and grauelly substance, the mine masters and mettall finers use to wash, and that which setteth downward, they burne and melt in the furnace. There is found likewise in the gold mines a kind of lead ore which they cal *Blutia*; for that the water that they let into these mines (as I said before) washeth and carrieth down withall certain little blacke stones streaked and marked a little with a kind of white, and as heavy they be in hand as the very ore of gold; and therefore gathered they be with the same ore, and laid in the paniers together therewith; and afterward in the furnace when the fire hath made a separation between them and gold, so soone as they are melted do resolve into the substance of the white lead or tin glance aforesaid."

If Pliny's observation that this variety of *plumbum candidum* is as heavy as gold could be relied upon, the view would be plausible that he was cognisant of platinum, but unfortunately in other places he gives evidence of great inaccuracy in this respect. So, too, there seems no good reason for considering the metallic *electrum* to be anything other than the natural or artificial white alloy of gold and silver. If there were any question as to whether platinum were known to the ancients, it would seem to be completely answered in the negative by the fact that no platinum object, no nugget, or grain of platinum has been found among ancient remains.

Soon after the introduction of platinum into Europe, no inconsiderable amount of work was done upon it by Watson, Scheffer, Lewis, Macquer, Marggraf, Bergman, Guyton de Morveau, and others. A few chemists, led by

* *Praeterea scito infunduribus, qui tractus est inter Mexicum et Darien, fodinas esse auricalchi, quod nullo igni, nullis hispanicis artibus, hac tenuis liquescere potuit.*

† As an instance of the views of Cortinovis may be cited the lines:—

Atrio cloyit ebur, trabibus solidatur ahenis
Calmen et in celas surgunt electra columnas,

from Claudian's Rape of Proserpina (Book 1., v., 164), where it is considered that *electrum* must mean platinum.

Buffon (*Obs. Sur. Phys.*, Rozier, 1774, lii., 322), cast doubts upon its elementary character. Buffon, when reading his History of Minerals to the Dijon Academy, held that platinum was an alloy of gold and iron, because it was attracted by a magnet, and, said he, if platinum be a metal there must be a second substance in nature attracted by a magnet. Von Milly believed that mercury was also present in the alloy, but Blondeau (*Obs. Sur. Phys.*, Rozier, 1774, iv., 154), professor of mathematics at Brest, showed the great improbability that platinum was anything other than a simple metal.

The first suggestion of a practical use for platinum seems to have come from Lavoisier's (*Annales de Chim.*, 1790, v., 137) observation of its value for laboratory utensils. Many efforts were made in the last decade of the eighteenth century to fuse platinum, or to get it into workable form. The first recorded success in this direction was that of Janetty,* a Parisian artisan, who melted platinum by alloying it with arsenic. It was also alloyed with lead and with bismuth, and then cupelled. Before 1800 l'abbé Rochon (*Ann. der Phys.*, Gilbert, 1800, iv., 282) wrapped grains of platinum in platinum foil, heated to redness and then hammered into an ingot. Moussin-Poushkin (*Chem. Ann.*, Crell, 1799, ii., 359) amalgamated platinum sponge with mercury and ignited in a muffle. Another process is described (*Phil. Mag.*, 1805, xxi., 175) of wrapping ammonium chloroplatinate in platinum foil, igniting, and hammering. In 1800 Knight (*Phil. Mag.*, 1800, vi.) published his process, which, with some modifications, was generally adopted, and remained in use till the metal was fused in the flame of an oxyhydrogen blow-pipe by Deville and Debray, more than half a century later. Knight's process consisted in heating platinum sponge in a nearly cylindrical but slightly tapering clay mould, and compressing it by a few hammer blows while in the furnace. This gave a coherent platinum which could be readily worked into an ingot. It was just about the opening of the century that the discovery of platinum in the Ural Mountains occurred,† and the supply being thus very materially augmented, the use of platinum in the laboratory became established. At the same time the study of the metal, from a chemical standpoint, led to the discovery of several other metals in the platinum ores. No less than five of the distinguished chemists of that day were working on the ore, with the special aim of separating out other metals which they had reason to believe were present. Palladium was first obtained by Wollaston; Collet Descotils was the first to publish indications of what we now know to be iridium, and Fourcroy and Vauquelin at the same time had this metal in hand in an impure form. The real discoverer of iridium should be recognised in Smithson Tennant, who not only separated it in purity, but also, at almost the same time, found in the same residues the element osmium, which seems to have been wholly overlooked by the French chemists. This chapter was closed a few months later by Wollaston's discovery of rhodium.

(To be continued).

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 2nd inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the chair. The special thanks of the members were returned to Dr. Ludwig Mond for his donation of £150 to the Fund for the Promotion of Experimental Research at Low Temperatures. Mr. G. Livesey was elected a member.

* *Chem. Ann.*, Crell, 1799, ii., 53. The name is also spelled Jeanny, Jeannetty, and Jannetty.

† Osann says (*Pogg. Ann. der Phys.*, 1827, xi., 311) that the first mention he can find of platinum in connection with the Ural Mts. is *Ann. de Chim.*, 1806, ix., 317, where Vauquelin speaks of a rumour that platinum has been discovered in Siberia. Osann could not trace the origin of this rumour.

A BIBLIOGRAPHY OF STEEL WORKS
ANALYSIS.

PART III.

By HARRY BREARLEY.

(Continued from p. 7).

CARBON (*continued*).

II. MODES OF RELEASING THE CARBON.

334. WEYL (C. N., v., 170).—The sample is made the + pole of a dilute HCl cell. C residue retains form of the sample. The process was previously suggested by Binks (C. N., v., 252).
335. OSMOND and WERTH (C. N., lvi., 35).—Re-heated steel attacked by Weyl's process leaves a C-Fe residue which is explosive or spontaneously combustible.
336. BLOUNT (C. N., lvii., 28).—Place borings on Pt plate in Cu solution, and suspend Cu plate thereover. Pass current so that Cu is removed from borings nearly as quickly as it is deposited on the upper plate. Escape of hydrocarbons thus obviated.
337. JUTSUM (C. N., xli., 17).—Solution in acid Cu solution leads to loss through evolution of hydrocarbons. Uses Weyl's process. Rubber to limit action to a portion of the rod. Filters through a straight tube plugged with muslin, sand, &c. KHO containing KNO_3 absorbs O, and unduly increases the result.
338. HARTLEY (C. N., xvi., 157). The gas liberated in Weyl's process may be such as was occluded by the iron.
339. BOUSSINGAULT (C. N., xvii., 267).—Powdered sample triturated with HgCl_2 , the HgCl_2 volatilised in a current of H, and dried residue weighed.
340. BOUSSINGAULT (F. I. S. I., 1886, 822).—Above process fails if more than 2 per cent Cr is present. Burns directly in O for thirty hours, or separation of Fe with Cl. FeW and FeMn are decomposed by the latter method.
341. FRESSENIUS (C. N., xxi., 167).—Volatilise Fe with Cl. The chlorine must be thoroughly dry. See also NAUMANN and MUDFORD (F. C. S., lxxii., ii., 209).
342. FRESSENIUS and HINTZ (C. N., lxi., 67).—Release the C from FeCr in a current of Cl, and burn with CrO_3 and H_2SO_4 . Graphite is determined on 10 grms. of the sample after digesting for weeks in oft renewed HCl.
343. WATTS (C. N., xiv., 279).—Discusses various modes of releasing the C. Volatilises Fe with Cl in fifteen minutes; describes apparatus and precautions.
344. GINTL (F. I. S. I., 1885, 589).—Chlorine should be passed over heated charcoal before being used to decompose the sample. When Fe is rich in manganese MnCl_2 envelopes the C residue, and necessitates the subsequent use of a Cl absorbent.
345. SCHUTZENBERGER and BOURGEOIS (C. N., xxxi., 207).—White cast-iron decomposed with CuSO_4 and an acid solution of Fe_2Cl_6 . The dried residue is a hydrate of carbon.
346. HOGG (C. N., lviii., 199).— Fe_2Cl_6 may be used to decompose the precipitated Cu, or initially with the heated copper solution. If used alone it causes loss of C. Silicious steels filter badly after standing.
347. ZABOUDSKY (C. N., i., 57).—Triturated in a pasty mass of cuprasodium chloride. Cu dissolved with Fe_2Cl_6 . Results of the colour process not comparable if metals are of different origin.
348. LANGLEY (C. N., lxii., 227).—International standards' work. Values from 1.02 to 1.15 per cent due to use of variously crystallised, acidified, &c., Cu solutions.
349. LANGLEY (F. I. S. I., 1891, ii., 321).—Above results due to tarry matter (pyridine) in cuprammonium salts. Recommends cuprapotassium salts.
350. DROWN (C. N., lxi., 5).—Acidified CuCl_2 promptly dissolves the iron and gives good results.
351. ARNOLD ("Steel Works Analysis," p. 29).—Has observed bubbles of gaseous hydrocarbons on using acid CuCl_2 (see 337).
352. DUDLEY and PEASE (F. I. S. I., 1894, i., 609).—Decompose Fe with cuprapotassium chloride. Duplicates agree to 0.005 per cent.
353. CARNOT and GOUTAL (F. C. S., lxxii., ii., 520).—Dissolve in cuprapotassium chloride in an atmosphere of CO_2 at 90°C . The moist residue is burned in oxygen.
354. CARNOT and GOUTAL (F. S. C. I., 1899, 521).—Blair's recommendation—dissolving in an HCl solution of cupro-potassium chloride—leads to loss at temperatures of 70° and over. Dissolving in faintly acid solutions at 95° in atmosphere of CO_2 rapid and reliable, except for Fe-Mn.
355. GALBRAITH (C. N., xxxv., 151).—The iron of chromium steels refuses to replace copper in the solution. (This is certainly not the case with cupro-ammonium chloride.—H. B.).
356. FÖRSTER (C. N., lxxii., 293).—Some kinds of wrought iron, and tungsten steels especially, evolve hydrocarbons on being dissolved in a quite neutral solution of cuprammonium chloride. Cuprammonium oxalate is suggested.
357. BRAND (F. I. S. I., 1887, ii., 364).—On separating the C with neutral or basic cuprammonium chloride, hydrocarbons are evolved, and tungsten steels are hardly worse than other kinds. The loss is very serious in low-carbon steels.
358. BRAND (F. C. S., lii., 866).—Decompose with HCl containing 24 per cent Br; destroy Br with ammonium oxalate. Use Ag spiral in combustion-tube to retain traces of Br.
359. MOISSAN (F. C. S., lxx., ii., 339).—Carbon is liberated from Al with HgCl_2 , Hg volatilised in H, and residue burned in oxygen (see 339). GONTHIERE (F. S. C. I. 1896, 830) uses cuprammonium chloride and wet combustion for Al and alloys.
360. BLAIR (C. N., lxiv., 66).—The HCl solution gives higher results than the neutral solution of cuprammonium chloride. Determines C by direct combustion and volatilising Fe in HCl. Cuprous chloride and anhydrous CuSO_4 , the most efficient purifiers of CO_2 . Repetition of Dudley's work (348).
361. GIRARD (F. I. S. I., 1899, i., 464).—Uses the cuprammonium chloride process for estimating C in FeCr or FeSi, for which the chromo-sulphuric acid process is not satisfactory.
362. BREARLEY (C. N., lxxiv., 63).—The dissolving sample is kept stirred by attaching the containing flasks, fitted with tubes, to the filter-pump. Vigorous shaking in a closed flask prepares a sample for wet combustion in fifteen minutes.
363. J. T. (C. N., lxxix., 169).—Accessory to above stirrer. Examination of the decomposition of steels with cuprammonium chloride and continued re-use of the same liquor.
364. RECKLINGHAUSEN (F. C. S., lxxii., ii., 19).—A shaking apparatus, driven by a small motor.
365. DUNSTAN and DYMOND (C. N., lxxv., 148).—A simple shaker, also driven by a motor.
366. WDOWISZEWSKI (F. I. S. I., 1898, ii., 555).—Dissolves the sample of steel in cuprammonium chloride with a shaking apparatus in five to eight minutes.
367. ZABOUDSKY (F. I. S. I., 1882, 357).—After treating white pig-iron with CuCl_2 the residue is $\text{C}_{12}\text{H}_6\text{O}_3$. It can be easily nitrated.
368. ZABOUDSKY (F. I. S. I., 1884, 297).—Liberated with CuSO_4 and NaCl; the dried residue contains

- varying percentages of C, according to the nature of the sample. Extremes, 65.5 to 71 per cent.
369. DONATH (Z. I. S. I., 1898, i., 493).—Examination of the C. residue. Nitro-derivative prepared therefrom identical with the substance giving Eggertz colour. All the combined carbon is not evolved on dissolving in dilute acid.
370. JUPTNER (Z. I. S. I., 1896, ii., 422). *Resumé* of the examinations made of the carbonaceous residue, gases liberated on dissolving steel and the state in which C exists in steel.
371. BÄCKSTRÖM and PAJIKULL (Z. C. S., liv., 420).—Volume and character of gases evolved on decomposing steel with acids; liquid hydrocarbons formed.
(To be continued).

COLORIMETRIC ESTIMATION OF VANADIUM.

By L. MAILLARD,

DURING the course of a series of physiological experiments, I was led to search for a simple, rapid, and convenient method for the estimation of small quantities of vanadium in the form of vanadic acid, or of an alkaline vanadate. These quantities being, as a rule milligrams only, and rarely amounting to as much as a centigram, mixed with large quantities of other salts, the ordinary gravimetric methods were not to be trusted. The same may be said of the volumetric methods based on the reduction of permanganate.

In chemico-physiological or toxicological researches it is of advantage to reduce manipulation to a minimum, and to effect the estimation of particular bodies without first attempting too high a purification, on account of the inevitable losses which render the relative error too high when working on such small quantities. The estimation must also be founded on a specific reaction, and not a common-place one, such as the reduction of permanganate, which might be influenced by the presence of all kinds of impurities.

I have utilised the well-known reaction of peroxide of hydrogen on vanadic acid described by Barreswill ("Note on a New Oxygenated Compound of Chromium," *Ann. Chim. Phys.*, 1847, Series 3, xx., p. 364), in the year 1847, and attributed by him to the formation of a pervanadic acid analogous to perchromic acid which he had just discovered. Peroxide of hydrogen, ether saturated with peroxide of hydrogen, ozonised ether, and ozonised essence of turpentine, when placed in contact with a strongly acid solution of a vanadate, develop a magnificent red colouration, which is still noticeable in a solution of 1/84,000th, as has been shown by G. Werther (*Journ. f. Prakt. Ch.*, 1861, vol. lxxxi., p. 195). This reaction is commonly used for detecting the presence of vanadic acid, but up to the present no author, to my knowledge at least, has proposed to use it for a colorimetric estimation. I therefore decided to find out whether the formation of pervanadic acid was total, and if the depth of colour could be made to serve as an exact measurement of the proportion of vanadium in the solution.

I first prepared a mother-liquor of metavanadate of sodium at one-tenth by means of the pure salt; I then made dilute solutions of metavanadate of 1/100th and 1/1000th, which served as standards of comparison. With the same solution of one-tenth I prepared dilute solutions containing various quantities of metavanadate, quantities which were actually known, but which I treated as unknown, and which I estimated by means of Duboscq's colorimeter by comparing them with the 1/1000th solution. The results always gave perfect concordance.

Into a graduated test-jar, very narrow and very tall, containing 25 c.c., I poured 10 c.c. of the neutral solution under examination. I then added, according to the quantity of vanadate, from 1 to 5 c.c. of pure hydrochloric

acid; then from 3 to 10 c.c. of ether saturated with peroxide of hydrogen, H_2O_2 . This ethereal solution is prepared in advance by leaving equal volumes of ordinary ether and commercial peroxide of hydrogen at ten volumes alone together in a flask. The acidity of this peroxide of hydrogen is of no account, since the operation must be carried out in a very acid solution. I then corked up the test-jar and shook it up well; immediately the aqueous liquid takes a more or less intense red colour, and the supernatant ether remains perfectly colourless. I then poured a little distilled water into the test-jar to bring the volume of the aqueous portion up to 15 c.c.

If we carry out the same operation on the titrated solution of metavanadate at 1/1000th, it suffices to place the two red liquids in the cells of the colorimeter, and to vary the thicknesses until the tints are equal. The amount of vanadium is inversely proportional to the thickness. To test this method of working, I made a large number of verifications bearing on the following points:—

1. The intensity of the colouration produced in a solution of metavanadate at 1/1000th is always the same. For example, three experiments were made with the following proportions of reagents:—

- A. 10 c.c. of metavanadate at 1/1000th + 2 c.c. of ether H_2O_2 + 0.5 c.c. of HCl.
- B. 10 c.c. of metavanadate at 1/1000th + 3 c.c. of ether H_2O_2 + 2 c.c. of HCl.
- C. 10 c.c. of metavanadate at 1/1000th + 4 c.c. of ether H_2O_2 + 1 c.c. of HCl.

These gave colours exactly identical.

2. The intensity of the colouration produced in a solution at 1/100th is exactly ten times greater than in solutions at 1/1000th; this is proved by numerous experiments. It is of great importance to use sufficient quantities of the reagents. I varied the volumes of ether and of HCl used for estimating a solution at 1/100th, of which the concentration x was assumed to be unknown, the solution at 1/1000th representing unity. I always took 10 c.c. of metavanadate at 1/100th, and obtained the following values for x :—

Metavanadate at 1/100th. C.c.	Ether saturated with H_2O_2 . C.c.	HCl.	x .
10	2	1	5.56
10	4	1	6.12
10	6	3.5	8.43
10	10	4	10.00

We thus see that to obtain a complete reaction it is necessary to take for 10 c.c. of metavanadate at 1/100th, 10 c.c. of ether saturated with H_2O_2 , and 4 c.c. of HCl. For 10 c.c. of metavanadate at 1/1000th, 2 c.c. of ether, and $\frac{1}{2}$ or 1 c.c. of HCl would be sufficient. The necessary amounts of reagents in any particular case can therefore be judged according to the supposed concentration, or by means of a preliminary experiment.

3. The coloured solution can be diluted at will without undergoing any variation in the total amount of colouring matter; we can thus always bring the tint to be estimated into the neighbourhood of the standard tint. This is of great importance for ensuring accuracy of estimation, which is the more sensitive as the tints to be compared are closer and more dilute. In this manner a solution at 1/100th treated as above, then diluted to 150 c.c. (that is the aqueous portion), gives, by comparison with the solution at 1/1000th taken as unity, the following results:—

Thickness in m.m. of the standard solution.	Thickness in m.m. of the solution diluted to $x/10$.	$x/10$.	x .
5.0	5.0	1.0	10.0
10.0	10.0	1.0	10.0
15.0	15.1	0.9933	9.933
20.0	19.95	1.0025	10.025

The accuracy of the estimation has no other limit than the sensibility of the eye to the red rays.

4. The colouration remains for hours, and even days, when sufficient ether is employed; the measurements may therefore be made without any undue hurry.

5. Finally, the colouration should not be influenced by impurities in the solution. I have not experimented with the salts of the heavy metals, which are easily got rid of, inasmuch as H_2S does not precipitate the vanadates, and that the precipitate formed by $(\text{NH}_4)_2\text{S}$ is soluble in excess of this reagent. But in toxicological research we may come across salts, such as nitre produced by calcinations, chlorides of potash and ammonia introduced by neutralisations, sulphates and phosphates coming from the phosphorus and sulphur of the tissues, and, finally, the alkaline-earthly salts. I therefore prepared a series of solutions containing all of these. In 100 c.c. I placed 0.1 grm. of metavanadate of sodium NaVO_3 , plus 5 grms. (that is to say fifty times as much), of each of the following salts:— KCl , NH_4Cl , K_2SO_4 , KNO_3 , Na_2HPO_4 , and CaCl_2 . The colorimetric method did not show any difference between these various solutions and pure metavanadate at 1/1000th.

This method will thus be of great use, as much through its accuracy as its rapidity. A standard of comparison is necessary, which may be either a solution of vanadic acid or a vanadate, carefully titrated once for all by the ordinary method. In practice it will be found to be advantageous to replace it by a suitably tinted red glass. This should not be too deep in colour, or the sensitiveness will be diminished. The most suitable tint seems to me to be that possessed by a solution of metavanadate at 1/1000th, viewed through a thickness of about 15 mm. A similar sheet of glass, previously accurately standardised, will dispense with the necessity for operating with the standard solution on every occasion; it will also reduce the manipulation to one-half, and will enable a complete estimation to be made in less than five minutes.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 12.

THE ESTIMATION OF SUCCINIC ACID IN FERMENTED LIQUIDS.

By J. LABORDE and L. MOREAU.

The methods now in use (Pasteur's method and Macagno's method) necessitate a great deal of manipulation and slow evaporations at the ordinary temperature, as there would be a loss of succinic acid at a higher temperature. This loss is due to a partial etherification of the acid by the glycerin contained in the liquors of fermentation. The author proposes the following method, which is quicker and quite as accurate as those already in use.

If we have to deal with liquors completely or nearly fermented, that is to say, not containing more than 1 per cent of sugar, we proceed as follows:—100 c.c. of the liquor are evaporated to dryness on the water-bath in a porcelain crucible having a flat bottom, together with 20 grms. of white sand previously washed with hydrochloric acid and calcined; the sand is well mixed with the residue, while the latter is still in a syrupy condition. The mass becomes hard on cooling. To facilitate its subsequent treatment, it is advisable to allow the mass to soften a little by exposing it to the action of the air for a few hours. It is then placed in a 250 c.c. matras, 100 grms. of No. 4 shot and 30 c.c. of ether are added. Agitation with the shot completely exhausts the mass after three fresh additions of ether, which is decanted each time on a flat filter. The ether is driven off, and the residue dissolved in a small quantity of boiling water, and a decimal solution of potash added in the presence of phenolphthalein. When the titration is finished an excess of potash is added, and we proceed to the saponification of the glyceric ethers; it suffices to evaporate the solution to dryness in a beaker on the water-bath, and to take up this

residue with water and titrate the free potash contained. The figure thus obtained for the total succinic acid is a little high; the error is due to a little volatile acidity, which is not completely driven off during the evaporation of the wine. To detect this volatile acidity it suffices to take up the liquor saturated with potash, to set free the volatile acid by tartaric acid, and to estimate it by distillation.

If we have to deal with incompletely fermented liquors, containing more than 1 per cent of sugar, we proceed as follows, remembering that too great a proportion of sugar interferes with the complete extraction of the succinic acid by the ether. The liquid is evaporated to a syrupy consistence, from 10 to 20 c.c. of alcohol are added, and some lead shot; we then introduce 50 c.c. of ether into the matras by small fractions, and thoroughly shake up the shot so as to cause intimate contact between the ether and the syrupy liquid, which is precipitated in the form of an emulsion. The ether is then decanted, a small quantity of alcohol is added, and the treatment with ether repeated. After three or four washings of this kind the whole of the succinic acid is removed. The ether is driven off by evaporation, and the alcoholic residue, placed in a flat-bottomed crucible with sand, is treated in the same manner as a liquor not containing any sugar.—*Ann. de l'Inst. Pasteur*, vol. xiii., p. 657.

ON THE AMMONIO-EARTHY PHOSPHATES.

By L. BARTHE.

In certain treatises, particularly that of Kippenberger ("Grundlagen für den Nachweis von Giftstoffen," 1897, p. 222), we may find the formation of ammonio-barytic phosphate given according to the following equation:—



Taking the preparation of BaNH_4PO_4 for granted, we thought, without ever having seen it doubted, that the preparation of the ammonio-strontic phosphate would not offer any difficulties, and as we described the best conditions for the preparation of the different phosphates a few years ago (*Comptes Rendus*, 1892, vol. cxiv., p. 1267), it appeared to be of interest to prepare the amino-strontic phosphates of the general formula $(\text{PO}_4)_2\text{Sr}_2\text{Am}_2$, or PO_4SrAm , or again $(\text{PO}_4)_2\text{SrAm}_2$.

We therefore acted successively with methylamine, ethylamine, aniline, and *p*-toluidine on chloride of strontium mixed with disodic phosphate, these bodies being used in equimolecular proportions. Under these conditions the white precipitate obtained, when washed, does not contain any sensible proportion of nitrogen, and corresponds to the formation of bistrontic phosphate $(\text{PO}_4)_2\text{Sr}_2\text{H}_2$.

	I. Grms.	II. Grms. $(\text{PO}_4)_2\text{Sr}_2\text{H}_2 = 367$.	Theory for $(\text{PO}_4)_2\text{Sr}_2\text{H}_2 = 367$.
Dried material ..	0.50	0.50	—
P_2O_5	37.50	37.50	—
P per cent ..	16.38	16.38	16.89
SrSO_4	0.505	0.5048	—
Sr per cent ..	48.13	48.12	47.68

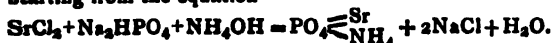
If we replace the amine by the hydrochlorate, and if to the mixture of the hydrochlorate of amine and chloride of strontium in concentrated aqueous solution we add disodic phosphate, we obtain a precipitate composed of phosphate of amine and phosphate of strontium. If we boil this mixed precipitate in several portions with distilled water, or if this same precipitate collected on a filter is thoroughly washed with cold alcohol, we obtain an amorphous, white, granular product hardly containing any nitrogen, and answering to the composition of the tristrontic phosphate, $(\text{PO}_4)_3\text{Sr}_3$.

	I. Grms.	II. Grms.	Theory for (PO ₄) ₂ Sr ₂ =452.5.
Dried material ..	0.50	0.50	—
P ₂ O ₅	32.00	31.00	—
P per cent	13.97	13.54	13.70
SrSO ₄	0.624	0.6249	—
Sr per cent	59.49	59.58	58.01

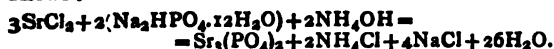
In the face of these results we may well doubt the existence of—



We therefore attempted to prepare this compound, starting from the equation—



Whatever method of mixing these compounds was adopted, or whatever concentration of solutions was used, we always obtained a white, colloidal precipitate, answering to the formula (PO₄)₂Sr₂. The tristrontic phosphate is really formed as the following equation shows:—



We have already obtained (*Comptes Rendus, loc. cit.*) this same tristrontic phosphate by pouring a cold ammoniacal solution of 90 grms. of disodic phosphate crystals into a solution of 100 grms. of crystallised chloride of strontium; both these solutions should be quite free from carbonates.

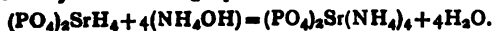
The preparation of the ammonio-strontic phosphate does not appear to have been effected by double decomposition. We have attempted to saturate a solution of monoammonic phosphate,—



which had been prepared by means of a saturated aqueous solution of strontia.

The addition of this latter solution immediately caused the appearance of a whitish magma, of which the chemical composition is very closely allied to that of the bistrontic phosphate.

Finally we have prepared the crystallised monostrontic phosphate. This compound was dissolved in a suitable quantity of water so as to facilitate its dissociation; ammonia was added in equimolecular proportion, as shown by the following equation:—



Immediately on the addition of the first drops of the alkaline solution a white precipitate is formed, which answers to the composition (PO₄)₂Sr₂H₄. No matter in what manner we have proceeded, we have been unable to obtain the ammoniostrontic phosphate.

There remained to verify the existence of the ammonio-barytic phosphate—



We have endeavoured to prepare this substance by taking advantage of the data furnished by the equation given by Kippenberger. As might be expected from the preceding results, we obtained bibarytic phosphate, (PO₄)₂Ba₂H₄.

	Grms.	Theory for (PO ₄) ₂ Ba ₂ H ₄ =466.
Dried material ..	0.5	—
BaSO ₄	0.502	—
Ba	0.2932	—
Ba per cent	59.04	58.80
P ₂ O ₅	28.0	—
P per cent	12.22	13.30

To sum up, with the exception of the ammonio-magnesian phosphate, NH₄MgPO₄, the other ammonio-earth phosphates do not exist.

To estimate the alkaline-earthly metal in phosphates, it is best to dissolve, at a boiling temperature, 0.50 grm. of the dried, pulverised phosphate in 5 c.c. of normal hydrochloric acid and 20 c.c. of distilled water. To this solution we add a few cubic centimetres of sulphuric acid diluted to one-fifth. This is kept at the boiling-point for a few moments; after cooling we add 20 c.c. of strong alcohol. Filter, after a few hours, and wash thoroughly with dilute alcohol. However long the washing may last, we generally find that there remains on the filter with the earthy sulphate a small quantity of phosphoric acid, which may be detected by means of molybdate of ammonia.

As for the estimation of the phosphoric acid, this can be done volumetrically by means of uranium in the washwaters, without the necessity of converting it into the ammonio-magnesian phosphate precipitate, after having taken the precaution of driving off the greater part of the alcohol by evaporation.—*Bull. Soc. Chim.*, Series 3, vol. xiii., No. 11.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 21st, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

A BALLOT for the election of Fellows was held, and the following were subsequently declared duly elected:—Messrs. James Ashton; George E. Battle, B.A.; Charles Berjen Brooke, jun.; Angelo Cantin; Ernest Owen Courtman; Richard H. C. Gompertz, B.Sc.; John Henry Gough; Henry Bertie Gritton; Percy John Hinks; Tajiro Ichioka; John B. Leathes, M.A., M.B.; Alexander Ernest McKenzie; Hugh McNair; Sidney Scrivener Napper; David Herbert Patrick; M. C. Nanjunda Row, B.A., M.B.; M. Goolab Roy; Oswald Silberrad, Ph.D.; Timothy A. Smiddy; Herbert Procter Smith; George Edward Tomline; John Traquair.

Of the following papers, those marked * were read:—

*89. "Researches on Morphine." I. By S. B. SCHRYVER and F. H. LEES.

The authors have found that the alcoholic hydroxyl group in morphine is readily replaceable, and they have prepared the compounds described below.

Chloromorphide, C₁₇H₁₈O₂NCl, prepared by the action of phosphorus trichloride on dry morphine. It melts at 190° with decomposition, and dissolves readily in chloroform and in methyl alcohol. The *hydrochloride*, C₁₇H₁₈O₂NCl.HCl, and *hydrobromide*, C₁₇H₁₈O₂NCl.HBr, have also been prepared. On treatment with acetic anhydride, the *monoacetyl* derivative, C₁₇H₁₇O(OC₂H₅O)NCl, is obtained, melting at 178–179°.

Bromomorphide, C₁₇H₁₈O₂NBr, melting with decomposition at 170°, has a very bitter taste and dissolves readily in chloroform and benzene. Its *hydrochloride* and *hydrobromide* crystallise with 1 molecule of water. Bromomorphide can also be prepared by the action of concentrated hydrobromic acid on morphine.

By treating chloromorphide with tin and hydrochloric acid, *desoxymorphine hydrochloride*,—



is obtained as glistening needles. The salt is laevorotatory; [α]_D²⁷ = –140.3°.

It was found that on heating chloro- and bromomorphide and their hydrochlorides with water, decomposition took place, bromomorphide giving the hydrobromide of a base isomeric with morphine; for this new base the authors propose the name *isomorphine*. Isomorphine and its derivatives are more powerfully laevorotatory than morphine and its corresponding deriva-

tives. The salts of isomorphine are much more readily soluble than those of morphine. Isomorphine melts at $246-247^{\circ}$, is readily soluble in methyl alcohol, but only very sparingly so in ether, chloroform, or benzene. Recrystallised from hot water, it is obtained as glistening needles. In methyl alcohol $[\alpha]_{D25} = -164.3^{\circ}$. For the hydrochloride, $C_{17}H_{19}O_3N.HCl$ $[\alpha]_{D25} = -150^{\circ}$; for the hydrobromide, $C_{17}H_{19}O_3N.HBr.H_2O$, $[\alpha]_{D20} = -127.2^{\circ}$ for the anhydrous salt. The methiodide, $C_{17}H_{19}O_3N.CH_3I$, melts with vigorous decomposition at 279° ; in aqueous solution, $[\alpha]_{D25} = -91.5^{\circ}$. Morphine methiodide also melts at 279° , but for it $[\alpha]_{D25} = -72.9^{\circ}$.

By treatment of isomorphine methiodide in aqueous solution with silver sulphate and barium hydroxide, a very alkaline solution of the hydroxide is obtained, which, dried in a vacuum, sets first to a deliquescent mass of fern-like crystals, and on further dehydration in a vacuum yields a solid which can readily be powdered. This substance dissolved in methyl alcohol does not react in the cold with methyl iodide, but does so readily on warming, giving a methiodide extremely soluble in water and in methyl alcohol, but only slightly so in ethyl alcohol. For it $[\alpha]_{D25} = 96.4^{\circ}$. This compound is not identical with codeine methiodide.

The determinations recorded in the following table were made with the free bases dissolved in methyl alcohol, and the salts (anhydrous) in water:—

Free base.	Hydrochloride.	Hydrobromide.
Morphine: $[\alpha]_{D23} = -130.9^{\circ}$ $[\alpha]_{D26} = -111.5^{\circ}$ $[\alpha]_{D20} = -100.4^{\circ}$		
Chloromorphide: $[\alpha]_{D27} = -375.2$ $[\alpha]_{D21} = -313.7$ $[\alpha]_{D19} = -268.6$		
Bromomorphide: $[\alpha]_{D25} = -65.6$ $[\alpha]_{D27} = +41.1$ $[\alpha]_{D25} = +39.5$		

Chloromorphide, bromomorphide, desoxymorphine, and isomorphine are all devoid of narcotic action. The significance of these facts and the relationship between morphine and isomorphine were also discussed.

*90. "On the Oxime of Mesoxamide and some Allied Compounds." By MARTHA ANNIE WHITELEY, B.Sc.

The compound formed by the action of nitrosyl chloride on malonamide (Tilden and Forster, *Trans.*, 1895, lxxvii., 490) appears to be the isonitroso-derivative, $NH_2OC \cdot C(NOH) \cdot CONH_2$. It dissolves readily in water, has an acid reaction, and melts with decomposition at 187° . It forms alkaline salts which are bright yellow in colour and are easily soluble in water. Its most characteristic reaction is the production of an intense purple colouration when neutralised with an alkali and a solution of ferrous sulphate added: this is due to the formation of a double potassium ferrous salt, $KFe(C_3H_4N_2O_3)_2$, which crystallises from concentrated solutions in minute prisms having the lustre of bronze. The acetyl derivative of the oxime resembles the original substance in appearance, and melts with decomposition at 190° . The ethyl ether is crystalline, and melts at $150-151^{\circ}$.

The oxime can be prepared by the action of nitrous acid on the aqueous solution of malonamide, and by the action of hydroxylamine on dibromomalonamide.

By the action of nitrous acid on the oxime, a crystalline, sparingly soluble compound is obtained, which melts with decomposition at about 215° . It contains the proportion of nitrogen corresponding to a pseudo-nitrol of the formula $CONH_2 \cdot C \begin{smallmatrix} NO \\ NO_2 \end{smallmatrix} \cdot CONH_2$. It does not respond to Lieberman's test, nor does it react as an oxime. By the continued action of nitrous acid, oxalic acid was the only solid product obtained.

The oxime is reduced by hydriodic acid to aminomalonamide, $CONH_2 \cdot CHNH_2 \cdot CONH_2$, the hydriodide of which crystallises in prisms.

The oxime of pyruvamide, $CH_3 \cdot C(NOH) \cdot CONH_2$, prepared by the action of ammonia on the oxime of ethyl pyruvate, melts at $176-177^{\circ}$, and closely resembles the

oxime of mesoxamide, but the alkaline solutions exhibit no decided yellow colour, whilst the colour produced by the addition of ferrous sulphate to the alkaline solution is reddish-brown. In attempting to prepare the oxime of pyruvamide by the joint action of ammonia and hydroxylamine on ethyl pyruvate, the chief product obtained was the oximidohydroxamic acid, $CH_3 \cdot C : NOH \cdot C \begin{smallmatrix} NOH \\ OH \end{smallmatrix}$, which is readily soluble, melts with decomposition at 143° , and is distinguished, not only by the cherry-red colour which is produced by the addition of ferric chloride, but by the production of an intense reddish-brown liquid and subsequent precipitate on the addition of ferrous sulphate to the alkaline solution.

Succinamide submitted to the action of nitrosyl chloride or of nitrous acid is attacked but slowly, yielding succinic acid, but no oxime or other derivative.

Attempts to prepare Piutti's β -oxime of the half acid ethyl succinic ester were not successful; this was apparently due to the spontaneous conversion of the β -compound into Ebert's α -isomeride. Cramer (*Ber.*, 1897, xxiv., 1204) represents the β -compound as the stable, and the α - as the labile isomeride.

*91. "On Dimethylpyrone, Tetramethylpyrone, and Orcinol Derivatives from Diacetylacetone." By J. N. COLLIE, F.R.S., and B. D. STEELE, M.Sc.

That dimethylpyrone is capable of acting as a basic substance, forming salts with acids, has already been clearly shown (Collie, *Trans.*, 1899, lxxv., 710). This work has been continued, and the basic properties of tetramethylpyrone have been investigated.

The sodium salt of dimethylpyrone reacts with methyl iodide, giving dimethylpyrone, m. p. 87° .—



When warmed with hydrochloric acid this compound loses water and is converted into tetramethylpyrone, $C_7H_8O_3(CH_3)_2 = C_7H_6O_2(CH_3)_2 + H_2O$. Tetramethylpyrone melts at 92° . It crystallises from water as a monohydrate, m. p. $63-64^{\circ}$: it forms a hydrochloride and a platinichloride, but appears to be less basic than dimethylpyrone. From the residues after separation of the dimethylpyrone, two other substances have been isolated:—(1) trimethylpyrone, $C_8H_{10}O_2$, m. p. 78° , which forms a platinichloride; and (2) a derivative of orcinol, $C_9H_{12}O_2$, m. p. 150° , possibly trimethyl-1:2:4-dihydroxy-3:5 benzene.

This orcinol derivative resembles mesorcinol in a very remarkable manner, but it is difficult to see how mesorcinol, which is 1:3:5-trimethyl-2:4-dihydroxybenzene, could be produced from dimethyl-diacetylacetone.

A second orcinol derivative, $C_9H_{12}O_2$, was obtained by treating the crude product of the action of methyl iodide on disodiumdiacetylacetone with hydrochloric acid. It melts at 105° , and in all its reactions resembles the compound melting at 150° .

Dimethylpyrone unites with sodium ethoxide to form a compound, $C_7H_8O_2NaOEt$, which on treatment with acids yields ethyl-diacetylacetone, $C_7H_8O_3Et$. This latter substance is very unstable, and easily decomposes into alcohol and dimethylpyrone when heated with acids.

*92. "Dehydracetic Acid." By J. N. COLLIE, F.R.S. Since the publication of a paper on dehydracetic acid (Collie and Le Sueur, *Trans.*, 1894, lxxv., 254), several reactions and the properties of various compounds of the acid have been investigated, all of which agree with the formula put forward by the author some years ago, namely, that dehydracetic acid is the lactone of tetracetic acid. The production of dehydracetic acid from triacetic lactone has been accomplished, as indicated by the following equation:—



Although neither acetyl chloride nor acetic anhydride react with triacetic lactone alone, if the lactone be boiled

with acetic anhydride and a little sulphuric acid or sodium acetate, dehydracetic acid is formed. The action of ammonia on the isomeric acid $C_6H_5O_4$ (obtained by Feist from the dichloride of dehydracetic acid) has been investigated, and an acid, $C_6H_5NO_3$, has been obtained identical with that previously isolated from the products of the action of heat on ethyl β -amidocrotonate.

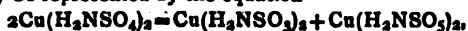
The production of dehydracetic acid from several of its dried salts has been studied. The dried sodium and silver salts treated with hydrogen chloride, and the dried lead salt with hydrogen sulphide, give dehydracetic acid.

The conductivity of a very pure sample of dehydracetic acid has been determined by Prof. J. Walker, who finds it to be 0.0009.

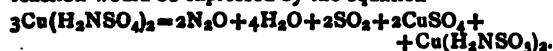
*93. "The Decomposition of Hydroxyamidosulphates by Copper Sulphate." By E. DIVERS and T. HAGA.

When a hydroxyamidosulphate is heated in solution with copper sulphate it is entirely decomposed. This takes place in such a manner as to throw much light on the nature of the obscure decompositions of these salts, as well as of the hydroximido sulphates and hydroxylamine salts when impure. No change is observed in the copper sulphate during the change it brings about, but of its alternate reduction and oxidation evidence is given, if, instead of the sulphate, cupric chloride be employed, as this is reduced to cuprous chloride.

The principal change caused by the copper sulphate may be represented by the equation—



the latter salt being represented by the actual products $Cu(H_2NSO_3)_2 = N_2O + H_2O + H_2SO_4 + CuSO_4$. Another change always takes place at the same time, being less marked when the temperature is low, and more so when the temperature is high. For example, a hydroximidosulphate resists the action of the copper sulphate until the temperature of the solution is over 100° , when it hydrolyses to hydroxyamidosulphate, 28 per cent of which decomposes so that all its sulphur becomes dioxide, its hydrogen water, and its nitrogen nitrous oxide. If one-third of the hydroxyamidosulphate decomposed thus, the reaction would be expressed by the equation—



Hydroxyamidosulphate decomposing under the influence of copper sulphate may give as little as 3½ per cent of its sulphur as dioxide. In all cases, the quantity of sulphur appearing as sulphate approximately equals that as amidosulphate. The production of any free nitrogen is still doubtful; it could only be formed when the sulphur as sulphate exceeded in amount that existing as amidosulphate.

*94. "The Degradation of Glycollic Aldehyde." By H. J. H. FENTON.

As glycollic aldehyde is now obtainable in a pure state (*Trans.*, 1895, lxxvii., 774), experiments are being carried on with a view of studying its relationships and properties. The author showed that by means of Wohl's reaction formaldehyde may readily be obtained from glycollic aldehyde. Glycollic aldoxime is obtained as a syrup from the interaction of the aldehyde and hydroxylamine in alcoholic solution, but it has not been obtained quite pure. When this syrup is mixed with acetic anhydride and sodium acetate, a violent reaction takes place, and the acetyl derivative of glycollic nitrile (b. p. 177°) is formed. This is identical with that obtained by Henry (*Comptes Rendus*, 1890, cx., 759—760) from formaldehyde and hydrogen cyanide. When this acetyl derivative is acted upon by the calculated quantity of ammoniacal solution of silver oxide, silver cyanide separates in a crystalline state, and a product is obtained which, with dilute sulphuric acid, yields abundance of formaldehyde.

*95. "Notes on the Chemistry of Chlorophyll." By LEON MARCHLEWICK, Ph.D., and C. A. SCHUNCK.

The authors first discussed the absorption spectrum of unaltered chlorophyll, and conclude (1) that this spectrum is characterised by three bands between the lines B and F, and three between F and K_β ; (2) that the bands exhibited by crude leaf extracts are caused by one chemical compound, chlorophyll, and not by several substances; (3) that no solution which does not exhibit this spectrum can be said to contain chlorophyll, no matter how it may have been prepared. Unaltered chlorophyll, by the action of hydrochloric acid, ought to give both phylloxanthin and phyllocyanin, the latter most certainly. The authors contend that Hartley's "blue chlorophyll" is not unaltered chlorophyll, but a derivative closely allied to alkachlorophyll, since its spectrum is quite different from that of unaltered chlorophyll, and gives with acids phyllotannin or its derivatives, but neither phylloxanthin nor phyllocyanin.

As to the several "chlorophylls," the authors corroborated the results of Hartley and Sorby's experiments, which tend to show the existence of a colouring-matter besides chlorophyll proper and the members of the xanthophyll group. They show, however, that Hartley's "yellow chlorophyll" is a mixture of this additional colouring-matter with members of the xanthophyll group, and that by removing the latter its colour is green; the name "yellow chlorophyll" is therefore unsuitable. Crude leaf extracts therefore contain two green colouring-matters, chlorophyll proper and another which is present only in small quantity; this exhibits a red fluorescence, and is characterised by an absorption band in the red which is more refrangible than that of true chlorophyll. The authors described a method by which chlorophyll could be obtained almost free from this green colouring-matter, and from the members of the xanthophyll group.

The action of bromine on phylloporphyrin and hæmatoporphyrin is very similar, and this is considered by the authors as an additional proof of the close relationship of these substances.

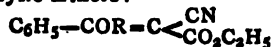
(To be continued.)

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

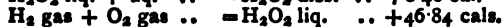
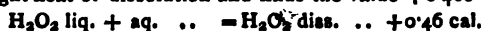
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 24, June 11, 1900.

The β -Phenyl and β -Benzyl α -Alcoyloxy- α -Cyanoacrylic Ethers:—



—A. Haller and G. Blanc.—The authors prepare these two ethers from their silver salts, and examine their properties.

Heat of Dissolution of Hydrogen Peroxide. Thermic Value of the Hydroxyl Function OH. Influence of Hydrogen and Carbon.—M. de Forcrand.—It has been found that it is just possible to obtain hydrogen peroxide almost in the anhydrous state by distillation under very reduced pressure. This fact proves that if the heat of dissolution of anhydrous H_2O_2 is positive, it is very slight. The author measures this slight heat of dissolution and finds the value +0.460 cal.



Further, he finds that for each of the two OH radicles, forming solid hydrogen peroxide, the value is +34.67 cal.

Direct Preparation of Crystalline Mercuric and Mercurous Iodide by the Wet Method.—F. Bodroux.—Crystalline mercuric iodide can be obtained by dissolving an excess of the amorphous substance in potassium iodide, or, better, in concentrated boiling hydrochloric acid. On cooling, the salt separates out in octahedra, or in quadratic prisms of a bright red colour. The yellow modification can be prepared either by sublimation or in the wet way by adding an excess of water to a solution of mercuric iodide in alcohol. The author observes that when a small quantity of ethyl or methyl iodide is left in contact with a large excess of a solution of a mercury salt at ordinary temperatures, mercuric iodide is produced. This experiment succeeds with the chloride, nitrate, and sulphate of mercury, but the finest crystals are obtained when the acetate is used.

The Impossibility of the Primary Formation of Potassium Chlorate obtained by an Electrolytic Method.—André Brochet.—The author's experiments show, in an absolutely clear and decisive manner, that during the electrolysis of alkaline chlorides, contrary to the hypothesis of Ostel, Haber and Grinberg, Foerster, Jorre, and Müller, the formation of the chlorate is never due to a primary action, but is always preceded by the intermediate formation of the hypochlorites, even in a very alkaline medium, and when the hypochlorite is not apparent.

Decomposition of Metallic Chlorides.—M. Echsner de Coninck.—The author continues his researches on the action of animal black on dilute aqueous solutions of HgCl_2 , CdCl_2 , Al_2Cl_6 , and SnCl_4 . Besides which, he makes new experiments with a dilute solution of Fe_2Cl_6 in water. This series of reactions, which are quite distinct from one another, are satisfactorily explained if it is allowed that animal black acts as a dialyser, and that the liquid contains, at any given moment, a modified oxide or hydrate of iron dissolved in hydrochloric acid. The animal black, which is in excess, takes up little by little the oxy-compound of iron, and hydrochloric acid passes into the filtrate. This explanation seems to conform with experiment, and also with the theoretical explanations which have been put forward by Graham, Debray, Krecke, and Kossel.

Hydrogenation of Acetylene in Presence of Reduced Iron or Cobalt.—Paul Sabatier and J. B. Senderens.—On comparing the actions of the various metals used in these experiments, copper, iron, and cobalt, on a mixture of hydrogen and acetylene, they are seen to present remarkable differences. The combination of two gases, with production of ethane, which accompanies a certain proportion of formenic carbides, is realised very easily by reduced nickel at ordinary temperatures, and also—but only when hot—by nickel. In the presence of excess of hydrogen, the amount of ethylenic carbides obtained is negligible. Copper acts less energetically, and gives a larger proportion of ethylenic carbides, whilst iron is the least active of all, and gives a very slight formation of ethane, whilst considerable quantities of free hydrogen are found in the gaseous product.

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* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

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Gases from Coal.—Will you inform me of any articles on the chemistry of gases obtained from the destructive distillation of coal, and also on the practical means of obtaining tar, ammonium sulphate, benzol, and potassium cyanide as by-products of the coking of coal?—J. W. E.

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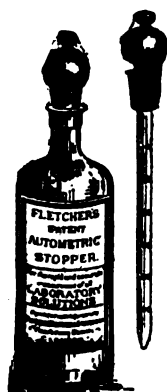
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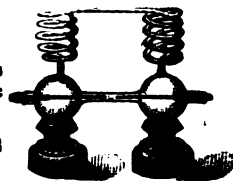
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2121.

ON RADIO-ACTIVE BARIUM.

By BÉLA V. LENGYEL.

AFTER the uranium rays of Becquerel had been discovered, and G. C. Schmidt had shown that the thorium compounds emit similar rays, Curie found in uranium ore (pitchblende) a radio-active substance whose radiation was about 400 times that of uranium. They predicted in this substance a hitherto unknown element, coinciding in its chemical reactions with bismuth, and which they called "polonium." They did not succeed in separating polonium from bismuth.

These same experimentalists, in conjunction with M. G. Bémont, isolated in the same year a more highly active substance from the pitchblende, in which they likewise predicted a hitherto unknown element that they called "radium." Radium possesses the chemical properties of barium, and is as inseparable from the latter as is polonium from bismuth. Demarcay examined the spectrum of radio-active barium, and found next to the intense barium lines a new line, which apparently belongs to radium. Mme. Skłodowska Curie determined the atomic weight of active barium, and found it eight units higher than that of inactive barium.

The existence of radium in radio-active barium compounds is established through the radio-activity, the spectrum, and the higher atomic weight.

F. Giesel likewise produced radio-active barium from pitchblende. He proceeded by a method quite different from that of Curie. Out of 1000 kg. of raw material he obtained 15 grms. of the preparation of radio-active barium. He also found some polonium combined with lead.

Finally, it may be remarked that Debierne had also isolated from pitchblende a very active substance, whose properties resemble those of titanium.

Many other experimentalists have worked at radio-active bodies; but their experiments were conducted almost exclusively with reference to the rays emitted by these bodies.

We thus know of radio-active bodies which have five different origins: the compounds of uranium, thorium, polonium, radium, and Debierne's substance resembling titanium. Uranium and thorium have been long known as chemically well-defined simple bodies; on the other hand, the three last-named are so far only hypothetical elements. Of these three hypothetical elements radium is the best known, as there are most accounts about it. The objective evidence of these accounts scarcely convinces one that radium is an existing element. For the existence of this element, two circumstances come principally into consideration from a chemical point of view, viz., the higher atomic weight of radio-active barium, and the spectrum of the same. Mme. Curie found the atomic weight of radio-active barium 8 units higher than that of the inactive, from which she concluded that radium must exist. But when we consider that radium, which so entirely resembles barium, must be divalent, and has as high an atomic weight as uranium, and, further, base our reckoning on the atomic weight of radio-active barium (145.8) found by Mme. Curie, it appears that the preparation from which the atomic weight was determined must have contained about 2 per cent radium chloride: that is a considerable quantity, and it cannot be assumed that such a quantity of a foreign element would not betray itself during the various chemical transformations.

Also, if a close chemical similarity between radium and barium is assumed, in consequence of which the two elements could not be distinguished and separated from each other by means of the usual analytical methods, it is yet difficult to accept radium as an existing element. Demarcay has examined the spectrum of radio-active barium and found in it only one line which was visible along with the intense barium lines, and which did not belong to barium. The spectra of calcium, strontium, and barium are of the same type; they consist of sharp lines and shaded bands; even the grouping of the lines and bands is alike. It was to be expected that radium, which is almost identically the same as barium, would show a similar spectrum.

Also, one must take into regard the fact that these new hypothetical elements are always found combined with other well-known chemical elements. M. and Mme. Curie found polonium combined with bismuth; Giesel with lead. Radium is combined with barium; Debierne's element with titanium. All these radio-active bodies have the same source, uranium pitchblende, from which they were separated by different analytical methods.

It can scarcely be maintained that chemical elements exist which are in no way distinguishable from other well-known ones, except in radio-activity; and it can scarcely be supposed that these elements, which can be transformed into various compounds, would in no single case give evidence of their presence in the chemical reactions but through their radio-activity.

Such considerations led me to examine experimentally the question as to whether the radio-active bodies contained new elements. After all the radio-active bodies known hitherto, which are said to contain a new element, were separated from the uranium pitchblende by analytical methods, I chose the synthetic method for the decision of the proposed question. For it is obviously clear that the question of radium being a chemical element must be answered in the negative as soon as it is found possible to transform ordinary inactive barium into the radio-active variety. At the same time this might be the synthetic production of radium.

My experiments carried out in this direction gave a positive result. It appeared that one can transform ordinary barium into a radio-active form which apparently possesses all the properties of radio-active barium observed by different experimentalists.

For the production of radio-active barium sulphate, the following should initially be noted:—Uranyl nitrate is melted together with 2 or 3 per cent barium nitrate, the nitric acid driven off as far as possible by sustained heating, and the remaining oxides fused in the electric arc. The fused mass is dissolved in nitric acid, and the solution evaporated, by which means a large portion of the baryta separates as nitrate. The hot solution is decanted from the crystals, diluted with three or four times its volume of water, and the radio-active barium sulphate precipitated with sulphuric acid. Only a small amount of sulphate is obtained in comparison with the quantity of barium nitrate used. I obtained, from 20 grms. barium nitrate, only 3 to 5 grms. sulphate, which is undoubtedly still largely mixed with ordinary barium sulphate.

The most favourable conditions for the production of radio-active barium are not yet determined. But the fact that a radio-active barium sulphate can be obtained as above described, seemed to me to be sufficiently important to publish in a preliminary notice.

I have so far prepared three compounds—radio-active barium sulphate, and from this the chloride and carbonate.

Radio-active barium sulphate is precipitated as a fine white substance, adhering rather readily to glass when the acid solution is treated with sulphuric acid or a sulphate. The precipitate, when well washed with hot water, dried, and heated, is white with a tinge of yellow, perhaps due to a trace of uranium. The substance was put into a little glass vessel, the bottom of which showed a faint gleam of light, and the glass placed on a sensitive

photographic plate, wrapped in black paper. After two hours the plate was developed, and a strong black mark appeared, corresponding to the transverse section of the glass. Hence the preparation was radio-active, and its activity increased after some days.

Radium rays are said to penetrate their metal sheets; my preparation of active barium sulphate emitted rays possessing the same property. One half of a copper coin was ground down to about one-third the thickness of the other half. The coin was laid between the vessel containing the barium sulphate and the sensitive plate, wrapped in black paper, and left there for three hours. After development, the plate was found to have remained white on the spot corresponding to the thicker copper layer, and had become grey beneath the thinner copper layer, of which there was an image surrounded with an intense black margin, indicating the action of the rays received direct on the plate beyond the copper coin.

Radium rays illuminate a barium platinocyanide screen; the rays of my substance exhibit the same property, though in less degree. The air becomes an electrical conductor by means of radium rays, and the same is the case with my prepared active barium sulphate. The carbonate and chloride prepared from the active sulphate were also active.

This is, so far, the limit of my researches. They do not nearly suffice to decide the question as to whether radium is an existing chemical element, or not; but those facts render doubtful the existence of radium. The question can only be settled by a searching examination, and I have in hand the preparation of the necessary material in sufficient quantity to be able to undertake this examination.

Chemical Laboratory of the University, Budapest II.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART III.

By HARRY BREARLEY.

(Continued from p. 19).

CARBON (continued).

III. ESTIMATING THE LIBERATED CARBON.

III.a. Weighing the Dried Residue.—

372. EGGERTZ (C. N., vii., 254).—Iron separated with iodine and dried residue weighed, or Fe separated with CuCl_2 and residue combusted. Graphite separated with HCl; residue dried, weighed, and ignited. Describes also the colour process and some of its limitations. Later (C. N., xlv., 173) uses iodine dissolved in ferric iodide for separating the carbon.
373. WITTSTEIN (C. N., xxii., 311).—Dissolve in $\text{CuSO}_4 + \text{CuCl}_2$, weigh dried residue. Loss on ignition = C (see 368).
374. BOUSSINGAULT (C. N., xxii., 317).—Separates with HgCl_2 (339); ignite residue at dull red; loss = carbon. Ignite in O current; loss = graphite.
375. HEID (C. N., lxxvi., 183).—After treating with alcohol and ether, the residue, dried at 120°C , is ignited; loss = C. Graphite estimated similarly.
376. DOUGHERTY (C. N., lxxx., 121, 146, 242, and 266).—Liberated carbon washed with hot dilute HNO_3 , dried, and ignited; loss $\times 0.675 = \text{C}$. Graphite similarly. Not available for FeMn.

III.b. Dry Combustion of the Residue.—

377. ABEL (C. N., vi., 133).—Dissolve in acid CuCl_2 (see 351), and burn residue with CuO . Liberate graphite with HCl, separate SiO_2 with KHO, dry, and weigh.

378. TOSH (C. N., xvi., 67).—Describes Regnault's (combustion with PbCrO_4 and KClO_3), Fresenius's (dissolve in H_2SO_4 , pass gases over CuO , and estimate C in residue), Wohler's (separation of Fe with Cl, and combustion of the residue), and Weyl's (334). Critical remarks on each process.
379. FORBES (C. N., xvi., 105).—Mainly like Abel (377). Dissolving for ten days.
380. PIESSE (C. N., xxviii., 198).—Decompose with warm acid cuprasodium chloride (see 354). Burn residue with CuO .
381. TURNER (C. N., lii., 15).—Decompose with cuprammonium chloride (see 483). The filter-tube is also the combustion-tube when resting on a small sheet-iron stand.
382. CLEMENCE (C. N., xlviii., 206).—Also burns the C in the (platinum) filtering-tube.
383. DEVILLE and TROOST (C. N., vii., 294).—Platinum tubes are porous to gases and vapours at high temperatures.
384. LANGLEY (C. N., lxii., 218, and *J. I. S. I.*, 1890, ii., 583).—"International Standard's" work. The CO_2 is purified with CuO , Ag, AgSO_4 , or CuSO_4 pumice, and CaCl_2 before being absorbed. An acid Cu solution gives higher results than a neutral one. Anhydrous CuSO_4 pumice soon becomes inefficient.
385. SHIMER (C. N., lxiv., 43).—Akin to Langley's paper. Direct combustion of the same samples. After liberal use of HCl all chlorides may be washed from the C residue.
386. DROWN (C. N., lxix., 4).—Oxygen passed over hot CuO before using. Cl retained by Ag, FeSO_4 , and anhydrous CuSO_4 . KHO bulb absorbs all the CO_2 (see 413), and CaCl_2 prolong all the moisture.
387. SHIMER (*J. S. C. I.*, 1899, 863).—The combustion is successfully made in an ordinary platinum crucible fitted with a water-cooled stopper. Apparatus serviceable for other operations generally requiring a tube.
388. DUPRÉ and HAKE (C. N., xliii., 69).—Absorb CO_2 in baryta water, convert BaCO_3 to BaSO_4 . BaSO_4 weighs 19.4 times as much as the original carbon.
389. GOOCH and PHELPS (C. N., lxxii., 194).—Estimation of CO_2 as in 388. Special apparatus to collect CO_2 . BaCO_3 washed under a protecting layer of xylene.
390. PHELPS (C. N., lxxiv., 65).—Estimates excess of $\text{Ba}(\text{HO})_2$ with iodine and arsenious acid.
391. RICHARDSON (*J. C. S.*, lxx., 469).—Apparatus for absorbing CO_2 in $\text{Ba}(\text{HO})_2$.
392. HANDY (*J. I. S. I.*, 1895, ii., 589).—Absorbs CO_2 in $\text{Ba}(\text{HO})_2$, and weighs BaCO_3 or titrates excess with H_2SO_4 .
393. MOISSAN (C. N., lxxii., 2).—Carbon is separated from Mo with Cl, and the residue combusted; or sample decomposed with acid in a stream of oxygen which passes over CuO into KHO. The graphite is determined in the residue.
394. BENNEVILLE (*J. I. S. I.*, 1895, i., 230).—C liberated with Cl, mixed with PbCrO_4 and $\text{K}_2\text{Cr}_2\text{O}_7$, and burned.
395. BLOUNT (C. N., lvii., 27).—Irregular results of Fresenius's process (378) due to formation of liquid hydrocarbons (see 369 and 371).
396. PHILLIPS (*J. I. S. I.*, 1887, ii., 372).—A solution of KHO in glycerin is used to absorb CO_2 .
397. BAKER (*J. C. S.*, li., 253).—Carbon containing moist oxygen gives rise to CO_2 when heated at 100°C .
398. STOKES (C. N., lvii., 150) uses asbestos instead of clay tiles, and an iron combustion tube closed with brass cap and leaden washer.
399. JERVIS (C. N., lxxvii., 5).—Arched asbestos cover for combustion furnace.
400. WHITE (C. N., xlv., 65).—An asbestos stopper made by pressing the moistened fibres into a mould.
401. DROWN (C. N., lvii., 223).—Uses a funnel with 20

wide a stem that the dried asbestos and residue can be pushed down it into the combustion tube.

402. BRENNEMAN (C. N., xlviii., 168).—A platinum cylinder fitted with a perforated disc acts both as filter and boat.
403. CARIUS (C. N., iii., 66).—The CO_2 is freed from SO_2 by passing through heated PbCrO_4 .
404. ETARD (C. N., xlvii., 178). Uses pumice soaked with $\text{Cu}(\text{NO}_3)_2$, and ignited to provide the CuO .
405. KNOP (C. N., iv., 13).—Collects rapidly evolved CO_2 in a rubber bag, and subsequently passes it through KHO (see C. N., lxxv., 241).
406. RUSSELL (C. N., xvii., 260).—Indiarubber absorbs notable amounts of CO_2 .
407. WILLIAMS (C. N., xliii., 69).—Black rubber is very pervious to CO_2 . See also KOBBE (Z. S. C. I., 1890, 1072).
408. BILGAT (C. N., lviii., 284).—The calorimetric bomb suggested as a combustion-furnace.

III.c. Wet Combustion of the Residue.—

409. ELLIOT (C. N., xix., 152).—Liberate C with CuSO_4 and acid CuCl_2 , and burn residue with CrO_3 and H_2SO_4 .
410. CAIRNS (C. N., xxv., 271).—Oxidation with CrO_3 used for graphite and for varieties of coal. See also Wiesner (Z. C. S., lxii., 1273).
411. PACKER (C. N., xxviii., 282).—Like 409. CO_2 passed through H_2SO_4 into the KHO . (See 332 and 414).
412. LANGLEY (C. N., lxii., 219).—"International Standards" work. A chlorine compound passes both AgSO_4 and anhydrous CuSO_4 into the KHO . Precautions devised.
413. PARRY and MORGAN (C. N., lxvii., 175).—Liberate C with neutral and acid cuprammonium chloride. A little CO_2 passes the Geissler bulb, and is retained by soda-lime (see 386).
414. SARNSTRÖM (Z. I. S. I., 1886, 1016).—The iodine carbon residue (372) is not of constant composition, and hydrocarbons are evolved during the dissolving. Hydrocarbons are evolved during combustion with CrO_3 ; these are burned to CO_2 with CuO .
415. GALBRAITH (Z. I. S. I., 1897, i., 567).—A process like Sarnström's.
416. KONINCK (Z. C. S., liv., 1341).— AgSO_4 added along with the CrO_3 prevents even a trace of chlorine being evolved.
417. BLUM (C. N., ix., 167).— AgSO_4 does not retain the chlorine if strong H_2SO_4 is used.*
418. AUCHY (Z. S. C. I., 1898, 604).—The KHO should have a sp. gr. of 1.40. Occasionally chlorochromic compound not oxidised by Langley's "pyro" (412) are formed. In the wet combustion of graphite CO is given off.
419. AUCHY (Z. C. S., lxxiv., ii., 534).—The error due to condensation of moisture on the KHO bulb cannot be allowed for by using a "dummy" bulb.
420. WIDMER (C. N., lxii., 274).—The C of organic bodies is not completely oxidised to CO_2 by CrO_3 and H_2SO_4 . Some CO is formed.
421. LUDWIG (C. N., xxv., 227).— CO is readily oxidised to CO_2 by saturated solutions of CrO_3 .
422. ALLEN (C. N., lviii., 21).— CO_2 is always evolved on heating pure H_2SO_4 and commercial CrO_3 .
423. PHELPS (C. N., lxxvi., 256).—Oxidises many organic bodies with CrO_3 , and after acting on Ludwig's suggestion, estimates the CO_2 . Estimates the combined oxygen also, by titrating the residual CrO_3 .
424. ANON. (Z. S. C. I., 1886, 617).—Combustion with

CrO_3 . Chlorine is absorbed by powdered metallic antimony,

425. LORD ("Metallurg. Anal.," p. 62).—The proper strength of H_2SO_4 in the mixture is from 50 to 70 per cent. Fused CaCl_2 usually contains CaO , and so can absorb CO_2 in the drying tube.

III.d. Volumetric Estimation of the Carbon.—

426. PARRY (C. N., xxv., 301).—Residue combusted with CuO in vacuum and CO_2 measured. Direct combustion of Fe gives low results.
427. IMBERT and COMPAN (C. N., lxxix., 267).—Carbon (lamp-black) is digested with H_2SO_4 and CrO_3 , and residual CrO_3 estimated with KI and $\text{Na}_2\text{S}_2\text{O}_3$.
428. J. T. (C. N., lxxx., 52 and 210).—Carbon liberated with Cu solution may be estimated by a modification of the above process, but not if Cr is present. Combined C is oxidised most rapidly by mixtures containing 50 to 70 per cent H_2SO_4 , and graphite by mixtures containing 90 per cent H_2SO_4 . Mixtures of $\text{K}_2\text{Cr}_2\text{O}_7$ containing 60 per cent or over of H_2SO_4 are slowly decomposed at 100°C .

(To be continued).

EIGHTEENTH ANNUAL REPORT OF THE COMMITTEE ON INDEXING CHEMICAL LITERATURE.*

The Committee on Indexing Chemical Literature respectfully presents to the Chemical Section its Eighteenth Annual Report, covering the nine months ending June 1, 1900.

Works Published.

"Index to the Literature of Zirconium." By A. C. Langmuir and Charles Baskerville. Smithsonian Institution, Washington City, 1899. 29 pp. 8vo.

This forms No. 1173 of the Smithsonian Miscellaneous Collections. The chronological list of references is followed by a Matter-index.

"A Bibliography of Steel Works Analysis." By Harry Brearley. CHEM. NEWS, lxxx., 233 et seq. (Nov., 1899).

The partial bibliography is confined to the contents of three English journals: CHEM. NEWS, Journ. Chem. Soc. (London), and Journ. Iron and Steel Inst.

The Committee also chronicles the publication of these foreign bibliographies:—

"Führer durch die gesammte Calcium-Carbid und Acetylen-Litteratur." Bibliographie der auf diesen Gebieten bisher erschienenen Bücher, Journale, Aufsätze in Zeitschriften, Abhandlungen und wichtigeren Patentschriften. Herausgegeben unter Mitwirkung von L. Ludwig. Berlin, 1899. 8vo.

This covers the industrial field as fully as the bibliography by Mathews does the scientific field, and both taken together are important for students of the subjects.

"Répertoire générale, ou Dictionnaire méthodique de Bibliographie des Industries Tinctoriales et des Industries annexes depuis les origines jusqu'à la fin de l'année 1896." Par Jules Garçon. Paris, 1899-1900.

The first volume of this extensive work contains a chapter on the sources of chemical bibliography, in which the author fully recognises the works issued under the auspices of this Committee and those published by the Smithsonian Institution. The author writes:—"America yields to no nation in the matter of bibliography; an

* Blum says, "According to Volhard, H_2SO_4 diluted with half its volume of water, does not attack AgCl . This, however, is a degree of dilution which never occurs in the combustion of carbon in irons." It has since been shown that sulphuric acid, even more dilute than Volhard's, effects the combustion most rapidly. (See 425 and 428).

* Advance proofs from the Proceedings of the American Association for the Advancement of Science, 1900, vol. xlix.

American devised the decimal system of bibliography, and Americans formed the Committee on Indexing Chemical Literature, of which the Reports, edited by Mr. H. C. Bolton, are found in the *Proceedings of the American Association for the Advancement of Science*, since 1883.

Reports of Progress.

Dr. Alfred Tuckerman has completed and sent to the Smithsonian Institution a Supplement to his "Index to the Literature of the Spectroscope," which covers the period from 1887 to 1899.

Dr. H. Carrington Bolton's *Second Supplement* to his "Select Bibliography of Chemistry," containing a list of 7500 chemical dissertations, is passing through the press; it will form a volume of the Smithsonian Miscellaneous Collections.

Mr. A. G. Smith, of Cornell University, is engaged on an "Index to the Literature of Selenium and Tellurium," which, it is expected, will be completed this summer.

Dr. Frank I. Shepherd, Secretary of the Cincinnati Section of the American Chemical Society, plans a bibliography of the *alkaloids*.

Mr. Frank R. Fraprie, of the University of Illinois, Urbana, Ill., writes to the Committee that he contemplates preparing an "Index to the Literature of Lithium."

The Committee chronicles the new method of indexing chemical substances used by M. M. Richter in his "Lexicon," and by the editors of the *Berichte der Deutschen Chemischen Gesellschaft*, in which the references to organic compounds are arranged under their empirical formulae; the Chairman of your Committee finds that Mr. Edwin A. Hill, of the U.S. Patent Office, has been engaged for more than two years in cataloguing chemical bodies under their empirical formulae for convenience of his office. Mr. Hill's system is adaptable to inorganic compounds as well as to those of carbon, and differs from the German plan in arrangement of the symbols, being much simpler. This method will be explained in print before long.

It is gratifying to note the increasing and continued interest in bibliography on all sides, and the Committee stands ready to encourage the movement in chemistry by practical assistance to those desirous of contributing to now considerable list of indexes. Address correspondence to the Chairman, at the Cosmos Club, Washington, D.C.

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CRYSTALLISATION OF STEEL AND IRON.

By JOHN PARRY.

THE freezing or solidification of fluid iron may very well be compared with the freezing of water. The tendency in both cases is to assume the crystalline form.

But the crystalline structure in the case of ice is not very apparent in the mass or block of ice; it presents an apparently amorphous structure, the crystals being so closely packed together.

Graham, however, states that the ice may exist in the colloid (non-crystalline form) and in the crystalline form. Colloid ice is formed in contact with water at 0° C., and it has the elasticity seen in all colloid or non-crystalline bodies: on the contrary, when water is frozen at few degrees under zero, it has a well-marked crystalline structure.

It would appear that, judging from recent research,

something like this occurs in manipulating and heating steel, more especially very soft metal, containing less than one-tenth carbon; and a study of the fracture of steel, variously heated, induced me to say, many years ago, that the freezing of water and the freezing (or solidification) of steel, were comparable phenomena—the water, of course, at the normal temperature of fluidity, and the steel at the usual point of fusion or fluidity.

As regards the apparent amorphous ice previously referred to, Prof. Tyndall has shown that, by passing a pencil of solar rays through a sheet of ice, as the ice fuses, well-defined crystals are gradually developed, and may be seen on the screen.

On cooling, of course the reverse phenomena may be observed: further it is clear that, if the heating or cooling is suddenly stopped at any given period, the mass, strictly speaking, cannot be in the original amorphous state (*i. e.*, fine-grained). So far as I am able to follow the vast mass of facts recently placed before us, bearing on the changes in the structure of steel, it seems probable that these are explainable by the same laws as govern the freezing of water, or the gradual fusion of ice under the varying conditions above referred to. There can, I think, be little doubt that brittle weak steel is thus at times manufactured from metal of irreproachable chemical composition.

Newport, Mon., July 10, 1900.

ON THE ACTION OF IODIDES AND OF HYDRIODIC ACID ON SULPHUROUS ACID.

By A. BERG.

THE recent publication of a paper by M. Péchard in the *Comptes Rendus* (vol. cxxx., p. 1188) has decided me to make known the results I obtained in my research on the action of iodides and hydriodic acid on solutions of sulphurous anhydride, a research commenced in 1899 and temporarily put on one side through stress of other work.

The yellow colouration taken by solutions of iodides in the presence of sulphurous acid, which I recognised not to be due to the presence of free iodine, caused me to suspect the formation of a dissociable compound of these two bodies.

M. Péchard's work has fully confirmed this suspicion.

What I want particularly to draw attention to in this paper is the ulterior reaction of the iodides and of hydriodic acid on sulphurous acid, a reaction which is not mentioned by the above-named author.

The yellow liquid, when left to itself, slowly deposits a powder formed of small spherical grains of sulphur, and sulphuric acid is found in the liquid. At the same time the colour of the liquid becomes gradually fainter, and finishes by disappearing completely. This reaction is so much the slower as the quantity of iodide or hydriodic acid used is less.

On the 25th March, 1899, I closed up, in a series of sealed tubes, equal quantities (20 c.c.) of a solution of sulphurous anhydride in boiled water, to which were added decreasing quantities of iodide of potassium. One of the tubes contained no iodide, and served as a check. The whole of them were left in a part of my laboratory which received direct sunlight for a portion of the afternoon.

	Numbers of the Tubes.					
	1.	2.	3.	4.	5.	6.
	Grm.	Grm.	Grm.	Grms.	Grms.	Grms.
Quantity of iodine..	0.1	0.5	1	2	4	8

On the 31st of March tube No. 1 commenced to deposit sulphur, and the colouration of the liquid became fainter and fainter, until, on the 5th of April, it had entirely disappeared.

Tube No. 2 did not commence to become cloudy until the 4th of April, and complete decolouration did not take place until the 21st of April.

Tube No. 3 became cloudy on the 12th of April, and colourless on the 17th of May.

Tube No. 4 became cloudy on the 22nd of April, and colourless much later (some time in September during my absence).

Finally, tubes Nos. 5 and 6 did not lose their transparency until the 4th of May for the first, and the 12th of June for the second, and are still to this day decidedly yellow in appearance.

In all these tubes a plentiful deposit of sulphur was found. On opening them, not a trace of sulphurous acid can be found in the colourless liquids. At the same time a scarcely perceptible odour of sulphuretted hydrogen can be observed, it is otherwise impossible to detect.

The solutions which are not completely decolourised still contain a small quantity of sulphurous acid.

The estimation of the deposited sulphur and of the sulphuric acid formed gives the following figures:—

	Numbers of the Tubes.		
	1.	2.	3.
Sulphur deposited	0.234 grm.	0.250 grm.	0.251 grm.
Sulphuric acid ..	1.487 grms.	1.490 grms.	1.380 grms.
Sulphur in the sulphuric acid ..	0.486 grm.	0.487 grm.	0.451 grm.

As for the check tube it did not undergo any change, and analysis showed that it contained 0.702 grm. of sulphur in the form of sulphurous acid.

We thus see that the precipitated sulphur and that of the sulphuric acid formed are practically in the proportion of 1 to 2, as required by the formula—



There are, however, no further reactions, for we find, either in the free state or in the form of sulphuric acid, the whole of the sulphur in the sulphurous acid used.

Light having appeared to take part in the phenomenon, on the 10th of April I prepared two similar tubes to No. 1 in the above series. I placed them in a continuous current of water in a light place, after having wrapped one of them up in a sheet of lead. On the 20th of April the tube exposed to the light was completely decolourised, while the other one was intact and showed no trace of any deposit. However, when left for a long time in the dark, it eventually lost its colour.

Light is not therefore a necessary factor in the reaction, but only acts by accelerating it. The blue and violet rays are the most active, the red rays behave almost the same as complete darkness.

Heat also acts as an accelerating agent, for of two tubes placed in darkness—one at the ordinary temperature and the other kept at 95°—the first took a long time to become decolourised, while the second lost its colour very rapidly.

Hydriodic acid behaves in the same manner as iodide of potassium, and is found unaltered at the end of the reaction, while in the case of the iodide, sulphate of potash is formed.

A small quantity of hydriodic acid or of iodide is thus likely to destroy, in the long run, large quantities of sulphurous acid; the yellow compound formed in the first place becomes decomposed, forming sulphur, sulphuric and hydriodic acids, or iodide, which may again react on a fresh portion of sulphurous acid.—*Bull. Soc. Chim., Series 3, xxiii., No. 12.*

Action of Acetylene on Cuprous Chloride dissolved in a Solution of Potassium Chloride.—M. Chavastelon. —The author finds that a prolonged action of acetylene on cuprous chloride, dissolved in potassium chlorate solution, causes the formation of colourless crystals amongst the yellow ones. These are orthorhombic in form.—*Comptes Rendus, cxxx., No. 24.*

DIAGRAM OF COMPOSITION OF IGNEOUS ROCKS.

By H. WARTH.

RECENT studies about the average chemical composition of larger numbers of igneous rocks in the aggregate have shown that figures obtained from any one hundred or more samples are very similar, in fact practically equal. (See A. Harker, "On the Average Composition of British Igneous Rocks," *Geological Magazine*, May, 1899, No. 5). This will be found also the case when comparing the

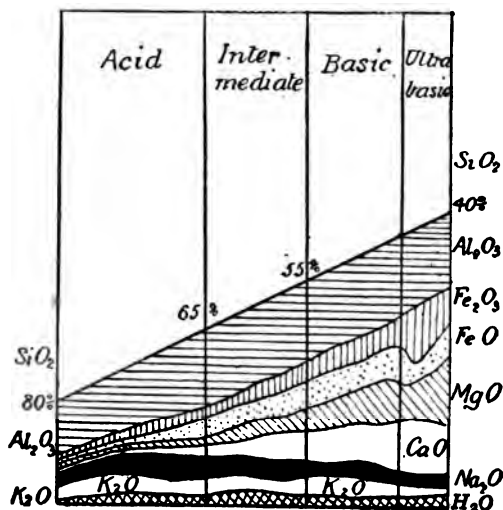


DIAGRAM-FIG. 1.

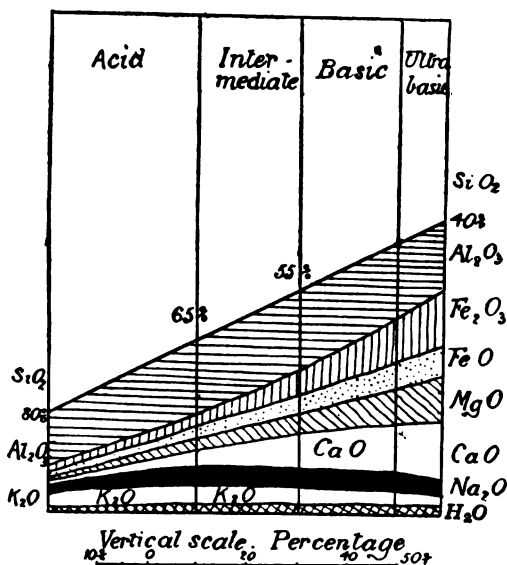


DIAGRAM-FIG. 2.

following average, which I calculated from the analysis of igneous rocks compiled by Roth in his "Petrographie der plutonischen Gesteine."

Now if it is of interest to discuss these average results, notwithstanding the wide discrepancies of many individual compositions, it may also be worth while to find out how on the average the compositions vary with the relative

acidity of the rocks. For this purpose I have arranged the bulk of Roth's samples in the order of acidity, and after calculating average compositions of eighteen groups of closely approaching acidity, I constructed from the figures the subjoined diagrams. The first diagram (Fig. 1) shows the actual results, the second (Fig. 2) shows them rounded off.

The percentage of silica varies from 80 to 40, and the usual limits between acid, intermediate, and basic rocks are indicated by the vertical lines on the diagrams. The diagrams show the percentage of the different bases which corresponds with the percentage of silica, indicated on the straight diagonal boundary-lines of the silica, which rise from the points marked 80 per cent on the left of each diagram to the points marked 40 per cent on the right of each diagram. As it would not be possible to give so many details on the diagram, small quantities of other acids have been reckoned with the silica and treated as such. These acids are titanic acid, phosphoric, carbonic, and in rare cases also sulphuric acid. In the case of the bases I also included the manganous oxide, and a few other rarer metallic oxides with the ferrous oxide. The total number of specimens which were calculated amounted to 428. About sixty-six of Roth's analyses were omitted, partly because they referred to strongly weathered specimens, and partly to exclude serpentines and some other more purely magnesian silicates. The specimens as given by Roth are from generally distributed localities, and the following are the compositions of extremes and of the total average in numerical figures:—

	Si	Al	Fe	Fe	Mg	Ca	Na	K	H
Most acid ..	79.7	11.4	1.1	0.3	0.7	0.9	2.6	2.5	0.8
Most basic ..	40.0	12.3	10.2	5.8	14.2	10.5	3.1	1.2	2.3
Average..	58.3	15.3	5.0	3.6	4.0	5.9	3.4	3.0	1.5

The diagrams show the alumina to be nearly constant, only slightly diminishing towards both extremes. The soda is also tolerably constant, diminishing slightly at both ends. The potash is decidedly stronger at the acid end, whilst the other oxides increase from left to right, so as to show quite a fan-like expansion from the acid end towards the basic end.

The diagrams show also that the arbitrary boundaries between acid, intermediate, and basic rocks are very suitably chosen.—*Geological Magazine*, vii., No. 432, June, 1900.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JUNE 30TH, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, July 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 200 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from June 1st to June 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter used in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 200 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The deficit in rainfall registered during the last three months has now given way to an excess. The rainfall at Oxford during June was 2.73 inches; the average fall for this month is 2.11 inches; this gives an excess for the month of 0.62 inch, making the excess for the first six months of this year 0.96 inch, or 8.7 per cent on the thirty years' average.

Our bacteriological examinations of 523 samples have given the results recorded in the following table; we have also examined 37 other samples, from special stand-pipes, &c., making 560 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 25 samples) ..	95
New River, filtered (mean of 131 samples) ..	8
Thames, unfiltered (mean of 25 samples) ..	1677
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 283 samples) ..	9
Ditto ditto highest	169
Ditto ditto lowest	0
River Lea, unfiltered (mean of 25 samples) ..	221
River Lea, from the East London Company's clear-water wells (mean of 34 samples) ..	11

Of the 460 samples of filtered water supplied to London, examined bacteriologically during the last month, less than 1 per cent has been found to contain above 100 and below 170 microbes per c.c. This demonstrates that the London supply more than maintains its normal good quality.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

THE EIGHTH GROUP OF THE PERIODIC SYSTEM AND SOME OF ITS PROBLEMS.*

By JAS. LEWIS HOWE.

(Continued from p. 17).

THE episode of the discovery of palladium throws a curious light upon the chemistry and chemists of that period. On May 12, 1803, Chenix read a paper before the Royal Society (*Phil. Trans.*, 1803, xciii., 290), stating that two weeks previously (April 29) he learned by a printed notice that a substance announced as palladium, a new metal, was offered for sale at the establishment of a well-known mineralogist. The printed notice (*Ann. de Chim.*, 1803, xlv., 333) opens: "Palladium, or new silver, has these properties among others which show it to be a new noble metal," and then follows the enumeration of eight characteristics of the metal, closing with the address where the new metal could be bought. Chenix goes on to say that the mode adopted to make known a discovery of so much importance without the name of any creditable person, except the vendor, appeared to him unusual, and not calculated to inspire confidence. Having examined a small sample, Chenix returned and purchased the whole supply. He then says: "We shall cease to wonder at what has been related by these chemists (Berthollet on affinity, and Hatchett on alloys), when we learn that palladium is not, as was shamefully announced, a new simple metal, but an alloy of platina; and that the substance which can thus

* An Address delivered before the American Association for the Advancement of Science, New York Meeting (1900), Section C.

mask the most characteristic properties of that metal, while it loses the greater number of its own, is mercury. Chenivix, however, on May 4, had written (*Ann. de Chim.*, 1803, xlii., 333) to Vauquelin that palladium really was a new metal, and Chenivix sent him a small specimen. A few days later another letter (*Ann. de Chim.*, 1803, xlii., 336) makes the claim that palladium is a platinum-mercury alloy. The editors of the *Annales* comment upon this, that from Vauquelin's experiments Chenivix is probably wrong. It is interesting to read the comments of Chenivix upon the masking of the individual properties of both platinum and mercury in palladium,* a correct moral drawn from a false premise. There was something almost prophetic in the observations of Chenivix, for there are in chemistry no elements whose properties exhibit such great variability when not pure as do those of the platinum group.

The still anonymous discoverer of palladium immediately offered, through *Nicholson's Journal*, a reward of twenty pounds sterling to any one who should manufacture even twenty grains of real palladium,† and many chemists entered into the discussion as to the elementary character of the metal. On June 24, 1804, three days after Tennant had made known the discovery of iridium and osmium, Wollaston read a paper before the Royal Society (*Phil. Trans.*, 1804, xciv., 419), acknowledging himself to be the discoverer of palladium. Wollaston had been engaged in an effort to obtain malleable platinum, and having precipitated his solution of the ore with iron, he found a part of this precipitate to be soluble in a mixture of hydrochloric acid with saltpetre, potassium chlorpalladate being formed. He at once concluded that palladium must be a simple metal, because there is "no instance in chemistry of a distinctly crystallised salt containing more than two bases combined with one acid," another correct conclusion drawn from a false premise. In his solution Wollaston found also indications of rose-coloured soluble crystals, which he attributed to another new metal, and to this he gave the name rhodium. This is the explanation of his curious method of making known his discovery of palladium: "On this and on other accounts I endeavoured to reserve to myself a deliberate examination of these difficulties, which the subsequent discovery of a second new metal, that I have called rhodium, has since enabled me to explain, without being anticipated even by those foreign chemists whose attraction has been particularly directed to this pursuit" (*Nicholson's Journal*, 1805, x., 204). It is possible from this that Wollaston himself had been led

to his work, in part at least, by the earlier observations of the French chemists.*

It was in 1803 that Collet-Descotils had read a paper before the Institute of France on the cause of the different colours which affect certain salts of platinum. The residue from the solution of platinum ores in aqua regia, and which was thought to be graphite, was still slightly soluble in aqua regia, and gave a reddish tint to the potassium chlorplatinate made from it. The metal derived from this red salt was found to be only partially soluble in aqua regia, and hence, beyond question, Descotils had in hand on several occasions a fairly pure iridium, containing, however, in addition to platinum, perhaps traces of rhodium. He was convinced that he had here a new metal, but he did not investigate its properties.†

The same day that Descotils spoke before the Institute, a second paper on the same subject was presented by Fourcroy and Vauquelin. After extracting the platinum from the ore by aqua regia, they fused the residue with potash, and then treated it with acid. These chemists noticed that the potash was coloured orange-yellow, but ascribed it to the presence of chromium. They thus missed the discovery of ruthenium, which was to be separated several decades later by Claus. They noticed also on one occasion that the black powder which consisted of iridium and allied metals was apparently volatile, not recognising osmium, the cause of the phenomenon. In their second memoir they had iridium fairly pure, and they, too, noticed the rose colour of the double salt, but did not investigate the cause, which is the presence of rhodium.

It was reserved for Tennant the following year to describe clearly the separation of iridium and osmium from the platinum residues, and to name the one from the many different colours through which its chlorides pass, and the other from the intolerable odour of its tetroxide, OsO₄.

This osmium contained ruthenium, but this was overlooked by Tennant, as it had been by Fourcroy and Vauquelin. A few years later Vauquelin (*Ann. de Chim.*, 1814, lxxxix., 241) notes that osmium solutions give a beautiful blue colour when reduced by zinc, this being a characteristic reaction, not of osmium, but of ruthenium. At a still later date Berzelius noticed the orange-colour of the fusion of ruthenium with potash and saltpetre, but he attributed it to iridium.

It was in 1828 that the next effort was made to add to the number of the platinum metals. Professor Osann, of the University of Dorpat, announced (*Pogg. Ann. der Phys.*, 1828, xiii., 287) the discovery of a new metal in the platinum residues. He obtained long reddish prisms, with high lustre, which were easily volatile, and which Berzelius pronounced to be new. He had only 0.3 grm., and never obtained any more. The metal in these crystals he named *ruthenium*. They may have been an impure mixture of the tetroxides of ruthenium and osmium. In the next volume of the *Annalen* (*Pogg. Ann. der Phys.*, 1828, xiv., 340) he transfers the name ruthenium to another new metal with a golden lustre, and at the same time he mentions two other new metals, *pluran*, named from pl (atina) and ur (al), which is not further described, and *polin*

* "The substance which has been treated of in this paper must convince us how dangerous it is to form a theory before we are provided with a sufficient number of facts, or to substitute the results of a few observations for the general laws of nature. If a theory is sometimes useful, as a standard to which we may refer our knowledge, it is at other times prejudicial, by creating an attachment in our minds to preconceived ideas, which have been admitted without inquiring whether from truth or from convenience. We can easily correct our judgment as to facts, and the evidence of experiment is equally convincing to all persons. But theories, not admitting of mathematical demonstration, and being but the interpretation of a series of facts, are the creatures of opinion, and are governed by the various impressions made upon every individual. Nature laughs at our speculations; and though from time to time we receive such warnings as should awaken us to a due sense of our limited knowledge, we are presented with an ample compensation in the extension of our views and a nearer approach to the immutable truth."—*Phil. Trans.*, 1803, xciii., p. 347.

† "Dec. 19, 1803, Editor *Nicholson's Journal*. Sir: As I see it said in one of your journals, that the new metal I have called palladium is not a new noble metal as I have said it is, but an imposition and a compound of platina and quicksilver, I hope you will do me the justice in your next and tell your readers I promise a reward of 20*g*., now in Mrs. Foster's hands, to any one that will make only 20 grains of real palladium, before any three gentlemen chymists you please to name, yourself one if you like. That he may have plenty of his ingredients, let him use 20 times as much quicksilver, 20 times as much platina, and in short of anything else he pleases to use: neither be nor I can make a single grain. Pray be careful in trying what it is he makes, for the mistake must happen by not trying it rightly. My reason for not saying where it was found was that I might make some advantage of it as I have a right to do. . . . I hope a little bit of whatever is made may be left with Mrs. Foster."—*Nich.* 7, 7, 1804, lxxv., 159.

* Since this Address was in type, I have received the interesting Presidential Address of Dr. Thorpe to the Chemical Society (London), in which this episode is treated quite exhaustively. What Wollaston's motive was in bringing his discovery to the notice of the scientific world in so extraordinary a manner, Dr. Thorpe says can only be surmised. Is not, however, in view of Wollaston's statement, the motive clearly apparent? He had found palladium; he was in pursuit of rhodium; he knew that at least four other distinguished chemists were also on track of new metals in platinum residues, and any hint, such as the publication of his work on palladium, would help them to anticipate him in the matter of rhodium, while if he kept silence some of them could not fail to discover palladium.

† Of this Descotils writes (*Ann. de Chim.*, 1803, xlviii., p. 125:—"On peut déjà conclure que la coloration en rouge des sels de platine est due à l'oxygénation d'une substance qui diffère du platine, et qui présente, lorsqu'elle est à l'état métallique, une grande résistance à l'action des acides."

(*polios*, grey), a grey metal of whose independent existence he seems to have some doubt. Polin was impure iridium with, perhaps, some ruthenium; pluran was quite possibly a mixture containing some ruthenium; the following year (*Pogg. Ann. der Phys.*, 1829, xv., 158), Osann acknowledged the metal to which the name ruthenium had been given to be a mixture of the oxides of titanium, zirconium, and silicon.

Fifteen years pass, and there appears at the University of Kazan, almost on the far eastern frontier of Russia, a chemist, Claus, who is destined to make greater contributions to the chemistry of the platinum metals, not only than those who had preceded him, but than any one of those who have lived in the nearly forty years since his death. Claus was fortunate in having at his disposal an almost unlimited quantity of platinum residues, from the stock which had accumulated at St. Petersburg during the period of the coinage of platinum. In spite of no considerable effort, I have failed to verify the tradition that the great mass of these residues was sunk in the Neva to prevent their use in debasing the coinage, and I am inclined to think that the greater portion was distributed to chemists, of which Claus received by far the largest share. In his first publication (*Bull. Akad. St. Petersb.*, 1845, iii., 38), Claus announces the discovery of a new metal, which he calls ruthenium, for the purpose of honouring Osann, whose ruthenium had failed to prove itself an element. It may be mentioned that Osann hardly appreciated the compliment, for he attacked Claus with considerable asperity, accusing him of claiming to discover what Osann himself had discovered. To an impartial critic Osann wholly fails to make out his case. For nearly twenty years Claus continued his work, and his greatest service was in definitely settling the position of the six platinum metals among the elements (*Bull. Akad. St. Petersb.*, 1860 [2], ii., 160). He was the first who clearly showed that these six metals belong in a group by themselves. Up to this time many had held that platinum belonged with gold, palladium with silver, and ruthenium with tin.* He then arranges the elements in three pairs and in two series,† as we find them in every table to-day. He also speaks of ruthenium and osmium being especially close to iron, an analogy hardly acknowledged, even after the periodic tables of different chemists had appeared. He adds that while platinum is not in the group with gold, it is closely related to it.

Since the time of Claus no new metals have been found in platinum ore or belonging to the platinum group, which have been generally acknowledged as elements. Several chemists have, however, found what they have believed to be new elements, but the quantity has generally been so small that their verification has been impossible. In 1852 Genth (*Proc. Acad. Sci. Phil.*, 1852, vi., 209; *Amer. J. Sci.*, 1853 [2], xv., 446) found two grains of a white metal in platinum from California gold. Its properties were unlike those of any known element.

In 1862 Dr. C. F. Chandler (*Amer. J. Sci.*, 1862 [2], xxxii., 351) described a new metal in native platinum from Rogue River, Oregon. This possessed properties very similar to those described by Genth, and Chandler concludes that his metal is probably identical with that found by Genth.

In 1869 Guyard discovered a metal in Russian platinum which he described ten years later (*Monit. Scient.*, 1879 [3], ix., 795), and called ouralum. It resembles platinum, but is softer, and has an atomic weight of 187.5. Its chief

difference from platinum is that when fused with potassium cyanide the melt is orange. This work of Guyard's has never been confirmed.

In 1877 Sergius Kern (*CHEM. NEWS*, 1877, xxxvi., 4, 93, 114, 155, 164; 1878, xxxvii., 65) announced the discovery of a new metal in platinum residues with atomic weight of 154, to which he gave the name of davyum. This has not only not been confirmed, but recently Mallet (*Ann. Chem. J.*, 1898, xx., 776) has gone over the whole ground with great care, and has shown that in all probability Kern's davyum is a mixture of iridium with rhodium and a little iron. Mallet obtained a residue in much the same way as Kern with similar properties and atomic weight, but proved it to be a mixture. This is the more significant since 154 would be about the anticipated atomic weight of a metal lying between the lighter and heavier metals of the group.

The great influence of one of the metals of this group upon the properties of another, even if present in but small quantities, has already been alluded to. It has been long known, and a very considerable series of experiments on this point is described by Claus.* Nevertheless it remains true that a good proportion of the workers on these metals have for a time at least supposed themselves discoverers of new elements. We may say, however, that not yet is there any reliable evidence of any new metal between the two series, that is, with an atomic weight of about 150; nor has there been any trace of *ekamanganese*, with its atomic weight of 200, and which some chemists would expect to find resembling the platinum metals in its properties.

It is by no means impossible that new metals may be discovered in this group; but the fact that in more than half a century no confirmed discovery of such has taken place, and that had it not been for a misinterpretation of reactions by which ruthenium was overlooked, we might say that it lacked but three years of a century since a new metal has been discovered, is not calculated to give us much encouragement. There does indeed seem, according to the periodic table, to be a place for three metals of atomic weight near 150, but it hardly seems probable that such occur in any of the known platinum ores which have been so thoroughly investigated, unless it be in extremely minute quantity. There is, however, always the possibility of the discovery of new platinum ores, differing in character from those now known, which, whether from the Ourals, or Colombia, or from the Pacific Coast, are approximately the same in composition.

We have seen that nearly half a century ago it was clear to Claus that iron, ruthenium, and osmium belonged in a group together. It was later easily recognised that cobalt, rhodium, and iridium furnished a second triad, while nickel, palladium, and platinum must also be grouped together. The analogies between the three metals of each of these groups is too patent to require discussion, though incidentally we shall have occasion to recur to it. When the elements were arranged in the first periodic tables, these metals did not fall into ordinary arrangement; as late as 1878 the atomic weight of osmium was considered greater than that of iridium, platinum, or even gold, while gold was given a weight less than that of iridium or platinum. Cobalt and nickel on one hand, and iridium and platinum on the other, were considered to have an identical atomic weight. The seeming impossibility of reconciling these nine metals with the periodic law is undoubtedly the reason why they were thrown out in a single group,—dumped into a chemical Gehenna as it were,—while the rest of the elements were reduced to orderly arrangement. Lothar Meyer, however, saw that there was a possibility that these elements also might be amenable to system, and under his direction Carl Seubert began the revision of the atomic weight of iridium

* *Loc. cit.* "Es ist nicht zu leugnen, dass in Beziehung einiger weniger Eigenschaften sich solche Analogien aufstellen lassen, allein ich habe Grund diesen nur einen geringen Werth beizumessen, und schliesse mich daher entschieden dem ersten Theile der Anschauungsweise der Verfasser an, indem ich, wie ich mich auch bisher ausgesprochen habe, die Platinmetalle alle für Glieder einer untrennbaren, wohl begründeten Metallgruppe halte."

† + end of the hori- zontal series.	Principal Series.	Os.	Ir.	Pt.	Vertical Series.	- end of the hori- zontal series.
	Secondary Series.	Ru.	Rh.	Pd.		

* C. Claus: "Beiträge zur Chemie der Platinmetalle," Dorpat, 1854, Chap. IV. "Modifikationen, welche die ursprünglichen Reactionen der einzelnen Platinmetalle durch Beimengungen der übrigen Metalle aus dieser Gruppe erfahren," p. 42.]

("Inaug. Diss.," Tübingen, 1879). This he found to be more than four units less than the figure formerly used, and now the order of these elements appeared to be iridium, gold, platinum, osmium. Three years later Seubert (*Ber. d. Chem. Ges.*, 1881, xiv., 865) revised the atomic weight of platinum, finding it lower than that of gold, and this work was confirmed by Halberstadt (*Ber.*, 1884, xvii., 2962), and by Dittmar and McArthur (*J. Soc. Chem. Ind.*, 1887, vi., 799). The only anomaly in these four metals now was in osmium, and this also was resolved by Seubert (*Ber. d. Chem. Ges.*, 1888, xxi., 1839), who found that the old value of Berzelius and Frémy was about eight units too high, and that, so far from having an atomic weight greater than that of gold, osmium in reality has the lowest atomic weight of the four metals. This revision was justly accounted a great triumph for the periodic law.

As with the other metals, so also much doubt existed as to the atomic weights of rhodium and ruthenium; but the work of Seubert and of Joly, while changing somewhat the older figures, confirmed the order given in Meyer's table. Much work has been done on palladium by Keiser and by Keller and Smith in this country, by Bailey and Lamb and by Joly and Leidié abroad. The figures for platinum and palladium represent a much greater degree of accuracy than those for the other four platinum metals. Indeed it must be said that little accuracy can be claimed for the present figures of rhodium and iridium, and certainly those of ruthenium and osmium cannot be depended on more closely than half a unit.

In the case of the three other metals of this group, the atomic weight of iron has been well determined, but is now being subjected to a most careful examination in the laboratory of Professor Theodore Richards. As was supposed a few years ago to be the case with iridium and platinum, so cobalt and nickel were thought to have the same atomic weight. Then Lothar Meyer showed that, judged by their properties, nickel should follow cobalt in the periodic system, and hence have the higher atomic weight. Revisions of these metals followed, but the more accurate the work the more probable it appeared that the atomic weight of nickel is below that of cobalt. It was suggested that nickel was quite possibly a mixture, and efforts were made to resolve it into its constituents. In this connection will be recalled the efforts of Gerhardt Krüss to decompose nickel, in which for some time he thought he had been successful, and christened the new metal gnomium. But like so many other aspirants for chemists' favour, gnomium proved to be but a mixture. The latest work on these metals, by Richards and Cushman and Baxter, far surpassing all that has previously been done, confirms the higher atomic weight of cobalt, and lends no support to the view that nickel is anything but a simple element.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

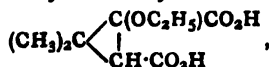
Ordinary Meeting, June 21st, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

(Continued from p. 23).

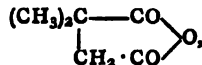
96. "A New Series of Pentamethylene Derivatives." I. By W. H. PERKIN, jun., JOCELYN F. THORPE, and C. WALKER.

When ethyl $\alpha\alpha'$ -dibrom- $\beta\beta'$ -dimethylglutarate,—
 $\text{CO}_2\text{Et}\cdot\text{CHBr}\cdot\text{C}(\text{CH}_3)_2\cdot\text{CHBr}\cdot\text{CO}_2\text{Et}$,
is treated with an alcoholic solution of potash, it is converted quantitatively into ethoxycaronic acid,—



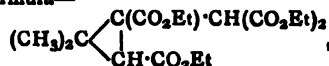
which separates from benzene in long needles melting at 136° ; the anhydride, made by distilling the acid, boils at $160\text{--}165^\circ$ (50 m.m.), and on boiling with water is reconverted into the acid.

When warmed with concentrated sulphuric acid, or when heated with hydrobromic acid in a sealed tube, the acid is converted into the anhydride of *asym*-dimethylsuccinic acid,—

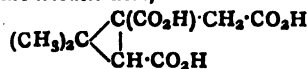


which melts at 30° , and on boiling with water yields *asym*-dimethylsuccinic acid melting at 139° .

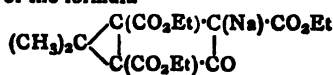
When ethyl dibromdimethylglutarate is condensed in alcohol solution with an equimolecular proportion of ethyl sodiomalonate, a 60 per cent yield of an ester of the probable formula—



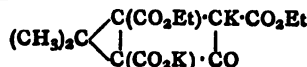
is produced; it is a thick liquid which boils at 234° (20 m.m.), and on hydrolysis with alcoholic potash is converted into the *tribasic* acid,—



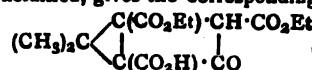
a colourless substance which separates from water in small needles and melts at 176° with evolution of aqueous vapour. When ethyl dibromdimethylglutarate is condensed with 2 equivalents of ethyl sodiomalonate in the presence of excess of sodium ethoxide, a yellow sodium compound of the formula—



is produced to the extent of 60 per cent of the theoretical amount; it is a remarkably stable substance, giving in alcoholic solution a deep red colouration with ferric chloride, and on hydrolysis in the cold with methyl alcoholic potash is converted into the yellow *potassium* compound—

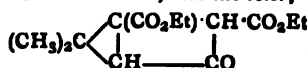


this, when acidified, gives the corresponding *acid ester*,—

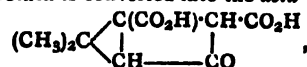


which crystallises from dilute alcohol in large prisms and melts at 75° .

On distilling this acid ester under diminished pressure, carbon dioxide is eliminated, and the *ester*,—

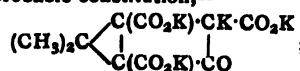


passes over at 201° (30 m.m.) as a thick oil which gives an intense violet colour with ferric chloride, and on hydrolysis with potash is converted into the *acid*—



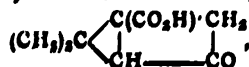
a colourless substance crystallising from concentrated hydrochloric acid in large prisms and melting at 179° ; it gives a red colouration with ferric chloride.

When the sodium compound mentioned above is treated with an equal weight of potash dissolved in ethyl alcohol, it passes into solution, and on boiling deposits a *potassium* salt, of the probable constitution,—



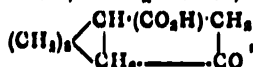
this, on being acidified, yields a mixture of two isomeric acids, which can be separated by re-crystallisation from dilute hydrochloric acid. The more soluble acid is identical with the acid mentioned above melting at 179°, the less soluble acid crystallises from dilute hydrochloric acid in needles melting at 154–155° with evolution of carbon dioxide; it gives an intense violet colour with ferric chloride; the first acid is called the α , the second the β -modification.

Both modifications, the β on heating above its melting-point, the α on heating with water in a sealed tube at 200°, are quantitatively converted into the acid,—



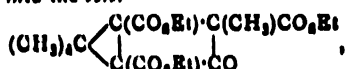
which crystallises from water in fern-like needles melting at 180° and subliming when heated, in long silky needles; the semicarbazone melts and decomposes at 225°, and the hydrazone at 217°.

On reducing the ketonic acid melting at 180° with sodium amalgam it behaves in a remarkable manner, the ketone group remaining intact and the trimethylene linkage being reduced, forming the acid,—



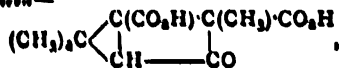
which separates from water in microscopic needles melting at 103°; the semicarbazone decomposes at 215°.

When the yellow sodium compound mentioned above is treated in alcoholic solution with methyl iodide it is converted into the ester—

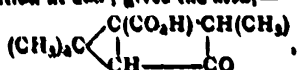


a colourless oil boiling at 210° (20 m.m.) and giving no colouration with ferric chloride.

On hydrolysis with methyl alcoholic potash, this ester is converted into a potassium salt which when acidified gives the acid—



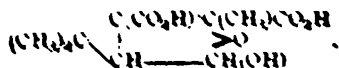
a colourless substance crystallising from benzene in lustrous plates melting at 146°; this, when heated in aqueous solution at 200°, gives the acid,—



which separates from water in glistening plates and melts at 134°; the semicarbazone decomposes at 230°.

If the potassium salt just mentioned be not separated, but the heating continued, it passes into solution with the formation of an acid characterized by being practically insoluble in dry ether, and which differs from the acid melting at 146° in containing 1 mol. of water of constitution. It separates slowly from water in large prisms melting at 437°, and when distilled under ordinary pressure passes over at about 270° as an oil which solidifies on cooling. This on boiling with water, is converted into an anhydrous acid which separates from water in needles melting at 126–131°, with formation of the anhydride.

The reactions of these compounds seem to indicate that they are stereoisomeric tetrane derivatives having the formula—



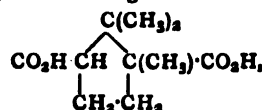
The investigation of these compounds, which are closely allied to several derivatives of the camphor and terpene series, is in progress, and experiments are being carried out in order to study the behaviour of other glutaric acids under the same conditions indicated above.

97. "Experiments on the Synthesis of Camphoric Acid. III. The Action of Sodium and Methyl Iodide on Ethyl Dimethylbutanetricarboxylate." By W. H. PERKIN, jun., and JOCELYN FIELD THORPE.

When ethyl dimethylbutanetricarboxylate (*Trans.*, 1899, lxxv., 900), dissolved in toluene, is treated with sodium and then with methyl iodide, a considerable yield of an ester is obtained which boils at 168–170° (18 m.m.), and on analysis gives numbers agreeing with the formula $C_{14}H_{22}O_6$. The method of formation of this substance clearly indicates that it must have one of the two following constitutional formulae:—



If the ester has the constitution represented by Formula I., it must be closely allied to, and will probably be easily converted into, an acid having the constitutional formula—



assigned by Bredt to camphoric acid.

On reduction by means of sodium amalgam, this ester is converted quantitatively into a syrupy hydroxy-acid, which on long standing shows signs of crystallising. The analysis of this acid and its silver salt shows that it has the formula $C_{10}H_{16}O_5$; it is therefore isomeric with camphoric acid. The authors are continuing the investigation of these substances.

(To be continued).

NOTICES OF BOOKS.

Dictionary of the Bibliography of the Dyeing and Allied Industries. ("Dictionnaire méthodique de Bibliographie des Industries Tinctoriales et des industries Annexes"). By JULES GARÇON. In three volumes. Vol. I. Paris: Gautier-Villars. 1900. Pp. 73.

THIS dictionary, or repertory, gives the titles of all papers, books, and documents published up to the end of the year 1896 which may be of interest to those engaged in the dyeing and allied industries.

Scientific and commercial work has increased of late years to such an extent as to have become international, and the technical papers published are so numerous that a special bibliography becomes indispensable for every industry.

Every subject bearing directly or indirectly on textile fabrics will be found in this repertory; it will therefore be of great use and assistance, not only to dyers, printers, and bleachers, &c., but also to all connected with textile fabrics or the chemical products used in their treatment.

The literature herein dealt with comprises about 2000 books and 110 periodicals; these latter consisting of 5000 volumes.

Electricity and its Applications. ("L'Électricité et ses Applications"). By DR. FOURVÉ DE COUERMELLES. Illustrated with 42 Figures in the text. Paris: Schleicher Frères. 1900. Pp. 185.

IT is a far cry to the discovery of the electric battery by Volta, but the gigantic applications of electricity now in use all spring from that occurrence. Although in its applications electricity may to a certain extent replace steam power, it is very unlikely that the steam-engine

will be ousted from its unrivalled position as the prime motive power. Waterfalls are useful in a few places, but there are not many of them, and their use is limited to the neighbourhood in which they are situated. It is, however, in its diversity of application that electricity commands our attention; it gives us not only the electric tram, crane, and lift, but the telephone, electric bell, and telautograph, while in its later developments we get Röntgen rays and wireless telegraphy.

The present volume deals with the subject in a popular historical manner without going deeply into the mathematical side of the subject, and is well worth reading.

CORRESPONDENCE.

THERMAL CENTRES OF STABILITY.

To the Editor of the Chemical News.

SIR,—In the very interesting article on "The Existence of Thermal Centres of Stability in Compounds," by Mr. Geoffrey Martin, occurring in the CHEMICAL NEWS of June 29th, the author states that in endothermic compounds "the atoms are held together in such a way that energy is stored up in the molecule," and in the decomposition of such compounds "work is done with, not against, the internal atomic forces."

Now although this explanation may be quite clear mathematically, when one tries to interpret it physically it seems hardly comprehensible. If the resultant of all the internal atomic forces is such that the atoms within the molecule *repel* one another, how could they possibly have been made to combine?

In the case of ozone, an example of an "endothermic" compound, is it not more reasonable to suppose that it is formed from its atoms with the evolution of heat? If we conceive a number of free oxygen atoms in a vessel, there would then be two courses open to them, either to combine forming ozone molecules with the evolution of a little heat, or to combine forming oxygen molecules evolving a greater quantity of heat. Thus, starting with oxygen molecules, we cannot convert them to ozone molecules, except by supplying energy to the system.

The only alteration suggested in the theory would be the substitution of the term "minimum heat of formation" for "maximum negative heat of formation," and certainly "negative heat" is a difficult physical conception.—I am, &c.,

H. W. HILL.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 25, June 18, 1900.

Formation of Nitric Acid during the Combustion of Hydrogen.—M. Berthelot.—Experiments on the combustion of hydrogen have to be performed under very different conditions from those under which carbon and sulphur were burnt, owing to the fact that the combustion of these latter elements takes place only at their surface, whilst the gaseous oxygen and hydrogen mingle throughout their entire mass, and combustion is almost instantaneous. The author performs his experiments in two ways: (1) by burning a jet of hydrogen in an atmosphere of oxygen, the combustion taking place in a progressive manner and at constant pressure; or (2), by previously mixing the two gases and exploding the mixture in a closed vessel by an electric spark, the combustion taking place at constant volume.

Combustible Gases in the Atmosphere. Air of Towns.—Armand Gautier.—The author examined the combustible gases in the atmosphere, in the streets of Paris, in summer and in winter. His experiments show that formene appears to be, as was suspected, the hydrocarbon which exists in the atmosphere of towns. He also proves the existence, to a slight extent, of methane in the air, but always mixed with free hydrogen and other hydrocarbons richer in carbon.

Action of Oxidising Agents on Alkaline Iodides.—E. Péchard.—The author has already shown that periodate of sodium, IO_4Na , is an energetic oxidising agent, capable of reacting on the alkaline iodides. He finds that a solution of this periodate decomposes sodium iodide in the cold. The solution thus obtained contains iodine, sodium iodate, and disodic periodate, the action being apparently represented by the equation—



This action is not instantaneous, the total quantity of iodine not being liberated until about an hour has passed. The next experiments were to find the action of ozone and hydrogen peroxide on potassium iodide. In each case analyses of the results were determined.

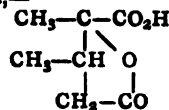
The Viscosity of Sulphur at Temperatures above the Temperature of Maximum Viscosity.—C. Malus.—Unsuitable for abstraction.

Selenides of Iron.—M. Fonze-Diacon.—The author prepares the compounds FeSe_2 , Fe_2Se_3 , Fe_3Se_4 , Fe_4Se_5 , and FeSe , corresponding to the sulphur derivatives of iron. He was unable to obtain the compound Fe_2Se by the reduction at a high temperature of iron selenides, this action only giving a mixture of iron and FeSe .

The True Atomic Weight of Boron.—G. Hinrichs.—M. Gautier has made two determinations of the atomic weight of boron from specimens prepared by M. Moissan. He finds that the ordinary atomic weight of boron is also the true weight. The application of the author's general method of calculation also bears out this statement, and proves that the specimen used must have been excessively pure. The atomic weight having 11.004 as a mean value, indicating that the true value should be exactly 11.

Action of Sulphurous and Hydrosulphuric Acids on Pyridine.—G. André.—In a former paper the author described a certain number of compounds of pyridine with formic, acetic, and propionic acids. He continues this research by considering the action of sulphur dioxide on pyridine, and was able—by direct combination of this gas with hydrogen sulphide, in presence either of pyridine alone or a mixture of water and pyridine—to prepare well-crystallised compounds, which are described.

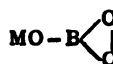
α - β -Dimethyl Glutolactonic Acids.—E. E. Blaise.—This preliminary notice relates to the α - β -dimethyl glutolactonic acids,—



which serves as the starting-point for the synthesis of the corresponding dimethylglutaric acids.

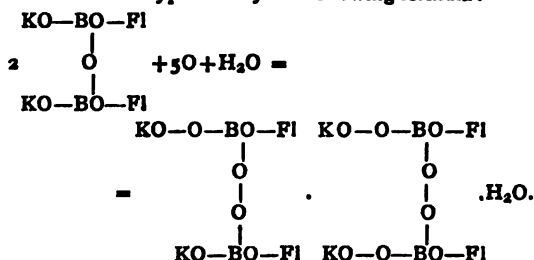
MISCELLANEOUS.

Fluohyperborates.—P. Melikoff and S. Lordkipan.—One of the writers has already published, in collaboration with M. Pissarjewsky, the results of a research carried out on the constitution of hyperboric acid, from which it follows that the salts of this acid may be given the general formula—



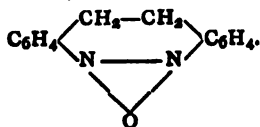
The experiments made, with a view to obtaining saline compounds corresponding with this formula, have not given very positive results. On the other hand, certain derivatives of hyperboric acid have the property of combining with the metallic superoxides. The authors used fluoborate of potassium, $B_2O_3 \cdot 2KFI$, which they prepared by Schiff and Sisti's method, by melting a mixture of boric acid and fluoride of potassium. On treating this salt, under previously determined conditions, with peroxide of hydrogen and caustic potash, they obtained fluohyperborate of potash which crystallises in prisms belonging to the rhombic system, and which forms alkaline solutions with H_2O . These latter, submitted to the action of heat, give rise to an energetic disengagement of oxygen. By the action of dilute sulphuric acid they form H_2O_2 . With the dried salt, which is fairly stable, concentrated sulphuric acid causes the formation of ozonised oxygen.

The relation between active oxygen, boron, and $\frac{K_2O}{2}$ in the salt may be expressed by the figures 1:25:1:1. By dividing the active oxygen between the boron and the potassium, we can represent the constitution and the formation of the hyperacid by the following formula:—



The saline compound obtained represents the potassic salt of fluopyroperboric acid, of which each H has been replaced by the radicle KO. The same substance may be prepared by adding with H_2O_2 on the potassic salt of orthofluoboric acid.—*Berichte*, vol. xxxii., p. 3349.

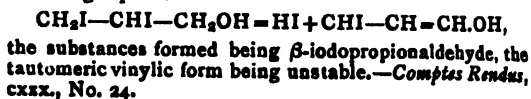
The Action of Sodium on Paranitrotoluene.—J. Schmidt.—By reacting at the ordinary temperature with metallic sodium on paranitrotoluene, a still more energetic reaction takes place than with nitrobenzene, but in this case we do not obtain a hydroxylamine derivative. If, for example, we add one equivalent of sodium to ether containing one equivalent of paranitrotoluene in solution, we are able to isolate parazoxytoluene from the product of the reaction. Besides this compound a brown sodic substance is formed, insoluble in ether and inflammable in the air, and which, according to the author, should be azoxydihydrostilbene,—



With two equivalents of sodium for one equivalent of paranitrotoluene there is produced, besides the above-mentioned brown sodic derivative, some parazotoluene and a small quantity of parazoxytoluene. The author proposes extending his research to other nitro and nitroso compounds, as well as to the oxides of nitrogen.—*Berichte*, vol. xxxii., p. 2919.

The Product of Decomposition of a Di-iodohydrine of Glycerin.—E. Charon and C. Paix-Séailles.—Di-iodohydrine of glycerin, $CH_2I-CHI-CH_2OH$, is easily prepared by dissolving iodine in slight excess of allylic alcohol. The mixture solidifies and crystallises easily. From this iodohydrine Hübner and Lellmann prepared a compound having formula C_3H_5IO , by loss of hydriodic acid, but they failed to determine the true nature of this

compound. The author's experiments lead him to believe that the di-iodohydrine decomposes according to the following equation:—



NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Oxide of Tin.—A correspondent in the United States asks for references to articles on the manufacture of oxide of tin.

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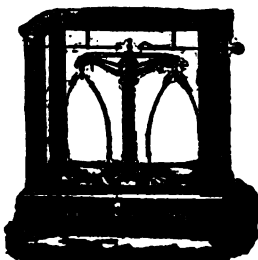
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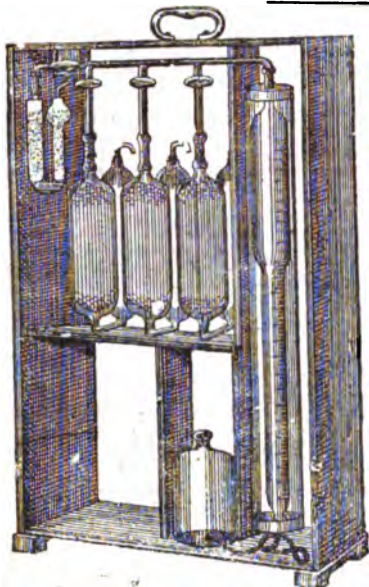
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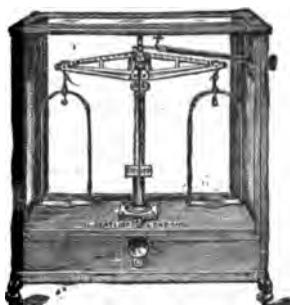
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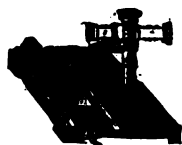
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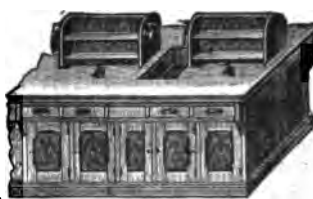
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THE EIGHTH GROUP OF THE PERIODIC SYSTEM AND SOME OF ITS PROBLEMS.*

By JAS. LEWIS HOWE.

(Continued from p. 33).

HERE we meet apparently one of those chemical mysteries which seem to baffle our attempts at solution. We are not permitted to doubt the correctness of the general principles of the periodic system, and yet here—and the case is perhaps not unique—two elements seem to have exchanged places. When we know *why* the properties of an element are a function of its atomic weight, we shall perhaps come to understand why the atomic weight of nickel is not greater than that of cobalt.

If the chemical study of these metals all supports the conception of their elementary nature, an examination of the spectrum of nickel and of cobalt, and particularly of iron, forces upon us the thought of the complexity of the atom. If each line of the spectrum, representing the vibration of a certain wave-length, is occasioned by a corresponding vibration of the atom, it becomes difficult for us to conceive of so many hundred simultaneous vibrations of a simple atom, a great share of which stand in no apparent harmonic relation to each other. It has been suggested that it is by a study of the spectroscopic portrayal of atomic vibration we may hope to gain the most complete knowledge of the dynamical character of the atom; but it must be remembered that with the spectroscope we study the motion of the atom at a high temperature, where vibration apparently overcomes in most instances chemical affinity; a knowledge of the atom at this temperature may give us no hint whatever as to the nature of the atom at lower temperatures, even as the spectrum of the same element may change with varying temperature. While we might thus conjecture that perhaps by some process of careful and refined fractionation, it might be possible to resolve iron into a series of meta-elements with nearly the same atomic weight, we are met by the fact that, complex though it is, we find not only the same spectrum for iron, whatever its terrestrial source, but that the spectrum of sideral iron, from meteorite, from sun, from star, gives us no evidence of any variation in the composition of iron. We are, I think, justified in concluding that the nine metals of the eighth group fulfil every definition of an element, and that they are just as much to be looked upon as simple elementary substances as any of those substances which we call elements; and further, that while refined determinations may change, to a slight extent, the atomic weight of some of these elements, especially those of ruthenium and osmium, we may expect the weight of these elements relative to each other, and hence their position in the periodic system, to remain unchanged. This, of course, carries with it the conclusion that in the periodic table an element may have an atomic weight slightly lower than that of the element which precedes it. I have discussed this possibility briefly elsewhere (CHEM. NEWS, 1899, lxxx., 74), and will only add that seeming exceptions to accepted laws, instead of overthrowing the law, often serve to broaden our conception of the law itself.

Before considering some of the compounds of the metals of the eighth group, attention must be called to

the phenomenon exhibited by several of these metals, and particularly by palladium, of condensing hydrogen and other gases upon their surface. The first observation in this connection seems to have been that of Sir Humphry Davy (*Phil. Trans.*, 1817, cvii., 77), who, in 1817, showed the Royal Society how a warm platinum wire, plunged into the vapour of alcohol or ether, or certain other inflammable gases, became incandescent, and continued to glow as long as kept in the vapour, causing an oxidation of the gas, and in some mixtures even an explosion. This phenomenon attracted great attention on the part of chemists, and many were the discussions over this lamp "without a flame," or Davy's "aphlogistic lamp" as it was called. It was soon after noticed by Edmund Davy (*Phil. Trans.*, 1820, cx., 108) that the platinum reduced from solution, now called platinum black, but then platinum suboxide, is especially active, and can oxidise alcohol to acetic acid. In 1823 Döbereiner announced (*Schweigger, J. für Chem.*, 1823, xxxviii., 321) that platinum black and platinum sponge, when held in a stream of hydrogen, ignite the gas, and that the hydrogen is absorbed by the platinum. This was the origin of Döbereiner's hydrogen lamp, regarding which he writes, under date of Aug. 5, 1823:—"You have doubtless guessed before this that I have already utilised this new observation (of the heating effect of the condensation of hydrogen) for the preparation of a new Feuerzeug and a new lamp, and that I shall put it to many other important uses" (*J. für Chem.*, loc. cit.). The interest which attached to this discovery of Döbereiner's can be judged by the fact that in the literature of the decade following are found over fifty references to the subject. Little attention was, however, paid to the similar action of palladium upon combustible gases, although the phenomenon had been noticed, until in 1868, half a century subsequent to Davy's first observation on platinum, Graham presented to the Royal Society his remarkable paper on the occlusion of hydrogen by metals (*Proc. Roy. Soc.*, 1868, xvi., 422), followed the next year by his papers on the relation of hydrogen to palladium (*Proc. Roy. Soc.*, 1869, xvii., 212), and additional observations on hydrogenium (*Proc. Roy. Soc.*, 1869, xvii., 300).

Graham's view that the hydrogen is present in solid form as a metal, and that the palladium saturated with hydrogen must be looked upon as an alloy, was received with considerable dissent. The work of Troost and Hautefeuille (*Comptes Rend.*, 1874, lxxviii., 686, 968; and 1875, lxxx., 788) tended to the view that the substance is a definite compound, Pd₂H. Against this is the fact that the conductivity of palladium is but slightly reduced by the occlusion of hydrogen. Calculations of the specific gravity of Graham's hydrogenium by Dewar gave the number 0.62, and the same figure is obtained for the hydrogen in hydrides of sodium and potassium, studied by Troost and Hautefeuille. The recent determinations of the specific gravity of liquid hydrogen by Dewar, however, show a figure only about one-ninth of the density of occluded hydrogen, so that the question as to the nature of the hydrogen condensed by the palladium and platinum remains still unsettled. The other metals of the group possess this property to some considerable degree, but much less than is the case with palladium and platinum. In this connection it is interesting to note that one of the earliest papers of Dewar was on the motion of a palladium plate, during the formation of Graham's hydrogenium (*Proc. Roy. Soc. Edinb.*, 1869, vi., 504).

Reference has been made to the natural grouping of the elements of the eighth group into three triplets, iron, ruthenium, osmium; cobalt, rhodium, iridium; and nickel, palladium, platinum. That this is a natural grouping is attested by a comparison of the compounds of these metals. However, in considering now some of these compounds, the evidence of this grouping is only incidentally presented; I desire chiefly to call attention to some of the more unusual of these compounds, especially with reference to problems which this group pre-

* An Address delivered before the American Association for the Advancement of Science, New York Meeting (1900), Section C.

sents, and to problems of other groups, suggested by the chemistry of this group.

The position of an element in the periodic system is, to a very considerable extent, determined by its oxides, and that too by its highest oxides, excluding the peroxides of the hydrogen peroxide type; a considerable number of these last have been studied especially by Melnikoff and Pissarjewsky of Odessa; but their character still presents many points of obscurity, and cannot be used with reference to the periodic law. The triplet iron, ruthenium, osmium, presents the highest oxides of the eighth group, and, as is the case with other divisions of this group, an increasing stability of the higher oxides with increasing molecular weight. The type of salts of the acid-forming oxides FeO_3 , RuO_3 , OsO_3 , occurs in this group, as in the positive series of the elements of the sixth and seventh groups; viz., CrO_3 , MnO_3 , WO_3 , UO_3 , MnO_3 . This type is not represented in the second or third triplet of group eight. Potassium ferrate, K_2FeO_4 , exists only in solution and is very unstable; potassium ruthenate, K_2RuO_4 , is stable when dry, but slowly decomposes when in solution; potassium osmate, K_2OsO_4 , on the other hand, has a very considerable degree of stability. Of the lower base-forming oxides, iron has not only the sesquioxide, Fe_2O_3 , and monoxide, FeO , but also several intermediate oxides, which may be looked upon as merely compounds of these two—such is magnetite. In the case of ruthenium, the sesquioxide, Ru_2O_3 , seems to be what one might call the normal base-forming oxide. The different conditions which occasion the formation of the lower oxides of osmium are ill known, though several different oxides seem to exist, as OsO , Os_2O_3 , and OsO_2 . Much more interest, however, attaches to the tetroxides of ruthenium and of osmium, RuO_4 and OsO_4 , which are the highest volatile oxides of any known element. The almost intolerable odour of osmium tetroxide incited Tennant, in 1803, to give its name to this element, while ruthenium tetroxide, first noticed by Claus, has, if not too concentrated, a rather fresh pleasant odour, with just a suspicion of the smell of ozone, due probably to the formation of ozone in the decomposition of the oxide. As far as physical properties go, these oxides, while solid at ordinary temperature, melt easily and can be distilled. Ruthenium tetroxide is, however, far less stable than the corresponding osmium oxide, for it decomposes slowly at ordinary temperature, and explodes with great violence if heated much above 105° . On one occasion Deville and Debray (*Ann. Chim. Phys.*, 1875, [5], iv. 537) attempted to distil 150 grms. of ruthenium tetroxide, and, when the temperature reached a little above 100° , the whole mass exploded with terrific violence, filling the laboratory with a dense sooty smoke. In a recent similar explosion in my own laboratory, occasioned by the contact of a little alcohol with ruthenium tetroxide, but happily on a much smaller scale than that of Deville and Debray, this black soot was found to be readily soluble in hydrochloric acid. This is unexpected, as all the anhydrous lower oxides of ruthenium are insoluble in acids, and from its method of formation the black substance could hardly be anything other than an anhydrous oxide or the metal itself. Osmium tetroxide is commonly known as osmic acid; but as a fact these tetroxides are neither acid-forming oxides nor are they peroxides in the ordinary acceptance of the term. When treated with an alkali we have a gradual reduction with formation of perruthenate and ruthenate, or of osmate.

Turning to the third triplet, we have the monoxide of nickel well characterised, and it is, we may say, the only well-characterised oxide of the metal, for though higher hydrated oxides of nickel exist, and perhaps anhydrous oxides also, their composition is not definitely known. With palladium and platinum also monoxides seem to exist and dioxides as well (PdO_2 and PtO_2). Platinum dioxide may be looked upon as being perhaps a very weak acid-forming acid. While nickel forms practically only the monoxide, NiO , and iron forms from choice, as we

might say, the sesquioxide, Fe_2O_3 , the intermediate metal cobalt, while forming most generally the monoxide, is easily oxidised to the sesquioxide, Co_2O_3 , and thus cobalt may be considered in its relations to oxygen as intermediate between nickel and iron. Similarly in the case of rhodium and iridium, there is a strong tendency to form the sesquioxide, so that this middle triplet is intermediate between the other two triplets of the group. As a whole, there is a large field at hand in a revision of the oxides of this group, especially those of the first triplet.

The same may be said even more emphatically of the sulphides. Those of iron, cobalt, and nickel are fairly well investigated, but of the remainder comparatively little is known except the somewhat exhaustive work of Schneider on the thioplatinates and thiopalladates. After a very considerable amount of work upon the sulphides of ruthenium, I have come to distrust about all that has been published, and to have nothing definite to add myself. The precipitates with hydrogen sulphide from ruthenium solutions (RuCl_3) contain apparently considerable free sulphur, but oxidise very rapidly with formation of sulphuric acid on drying, making their composition very difficult of determination. From ruthenate solutions a sulphide is precipitated which seems to have the formula RuS_2 , but there is no assurance that a part of the sulphur may not be free and not combined.

Of all the compounds of the metals of the eighth group, by far the best investigated are those with the halogens, and upon our knowledge of these rests the greater part of our chemical knowledge of the platinum metals. Yet here again our knowledge is wholly inadequate. If we except the work done under Wöhler's direction, by Oppler and Birnbaum on the bromides and iodides of iridium, that by Topsøe on the bromides and iodides of platinum, we may say that very little is known of any haloids of this group except the chlorides. In some instances, as with ruthenium, even the chlorides are very unsatisfactorily known. Of nickel we know only the bichloride NiCl_2 ; of cobalt the only stable chloride is the bichloride, CoCl_2 ; but the trichloride, CoCl_3 , seems capable of existence in solution; of iron, the ferric chloride, FeCl_3 , is the stable compound, into which the ferrous chloride, FeCl_2 , is readily oxidised. Here again the intermediate position of cobalt is apparent. There is a strong tendency on the part of all these chlorides to form double salts, of which we have examples in $\text{K}_2\text{FeCl}_4 \cdot 3\text{H}_2\text{O}$, K_2FeCl_5 , and Rb_2FeCl_6 . These salts seem to be broken up in solution, and the chlorine can be precipitated by silver nitrate. Turning to the halogen compounds of the platinum metals, we find double salts of a very different character. The common types for platinum and palladium, for example, are K_2PtCl_4 and K_2PtCl_6 . This latter type seems also to be known for all platinum metals except rhodium. Osmium, iridium, and rhodium present also the type K_2OsCl_6 , while ruthenium and rhodium also form salts of the type K_2RhCl_5 . The most important feature of these salts is that they are not decomposed when dissolved in water, silver nitrate precipitating not silver chloride alone, but the double chloride of the metal and silver; that is, for example, when K_2PtCl_6 is dissolved in water it is electrolytically dissociated, K being the positive ion, while the negative ion is the group PtCl_6 . The platinum metal then in these salts is a part of the negative ion. Double salts of this class are, of course, well known, but nowhere are they developed to the same extent as in the eighth group, indeed double salts of several acids are found among no other metals. The question may be fairly raised among the platinum metals as to whether there is any salt which is electrolytically dissociated giving the platinum metal as the positive ion.

The chlorides of ruthenium furnish an instructive illustration of the difficulties which may arise in getting complete knowledge of chemical facts as to what would naturally be considered simple substances. As we have seen, Claus discovered ruthenium in 1844. He obtained two chlorides or rather double chlorides, the one K_2RuCl_5 ,

and the other K_2RuCl_6 , the former corresponding to the type found in osmium and rhodium, the latter that found in platinum and palladium as well as iridium and osmium. In describing this latter salt, Claus shows that it was in the hands of Berzelius and probably in a pretty pure state, but that that chemist thought it to be an iridium salt. Berzelius was not convinced, and in the "Handwörterbuch der reinen und angewandten Chemie," edited by Liebig, Poggendorff, and Wöhler, the work of both Berzelius and Claus is given. This was naturally somewhat irritating to Claus, and he writes (*Bull. Akad. St. Petersb.*, 1860, [ii.], i., 108), "Even if reverence for the great authority of the great chemist (Berzelius) should seem to justify such a course, regard for the truth of science should not have permitted it." The subject was now dropped, and for a third of a century no one had worked upon this chloride, when Professor A. Joly, of the l'Ecole Normale, began his study of the platinum metals, and much of the work of Claus upon ruthenium was revised. Now it appeared that not only Berzelius, but also Claus himself was mistaken, and what he had taken for a chlor-ruthenate, K_2RuCl_6 , was in reality a nitroso-chlororuthenate, K_2RuCl_5NO . My own work of a little later date upon the chlorides of ruthenium abundantly confirmed this. Many efforts were made to prepare a tetrachloride of ruthenium, but it proved elusive. It may be noted in this connection that the caesium and rubidium nitrosochlorides exist in an anhydrous as well as in a hydrated form, and while the very easily soluble hydrated salts lose their water on warming the solution, the very slightly soluble anhydrous salt being precipitated, the reverse change is seemingly impossible, as the anhydrous salt will not take up water and pass back into the hydrated form.

The history of the higher chloride of ruthenium is not yet completed. Within the past year Professor Ubaldo Antony and A. Lucchesi, of the University of Pisa, have described (*Gazz. Chim. Ital.*, 1899, xxix.) the preparation of the real tetrachloride, K_2RuCl_4 , which, like the corresponding salts of platinum and the other metals of the group, crystallises in octahedra. I have more recently, by methods similar to those of Antony, prepared the caesium salt, Cs_2RuCl_4 , which also crystallises in octahedra and corresponds to Antony's salt; and I have also been fortunate enough to obtain a new salt of a rare type, lying intermediate between the tetroxide and the tetrachloride, $Cs_2RuO_4Cl_4(2CsCl, RuO_2Cl_2)$. Now a question may arise here as to whether Claus was, after all, wrong in believing he had the tetrachloride. As the salt was commonly made by Claus, by the action of nitric acid, it was, without question, a nitrosochloride, and his description corresponds completely; but Claus adds in a foot-note (*Y. Prakt. Chem.*, 1846, xxxix., i., 96) that it is also possible to prepare the salt by heating the trichloride with potassium chlorate and hydrochloric acid. This could not give the nitrosochloride, but while under these conditions the tetroxide is usually formed, it is possible that the tetrachloride may also have been formed. In another place he speaks of making it by the action of hydrochloric acid on potassium ruthenate, but in the presence of salt-petre. Except for this latter salt, this is the method of Antony, but usually at least it gives the trichloride. The salt which Claus generally describes is the nitrosochloride, but in one place (*Bull. Akad. St. Petersb.*, 1860, [ii.], i., 105) he says the salt seems to be dimorphous, for after crystallising out the common prismatic crystals (of the nitrosochloride) he, on one occasion, obtained large regular octahedra, isomorphous with tetrachlorides of the other platinum metals. As the molecular weight of the nitrosochloride is almost the same as that of the tetrachloride, and as the chlorine seems to have been generally estimated by loss, analysis would reveal no discrepancy, but in one case, at least, the chlorine was directly determined, and these figures can be accounted for only on the supposition that in this case it was a tetrachloride which was analysed, so that it would seem possible that Claus

actually formed the tetrachloride, although he did not distinguish it from the nitrosochloride. Even now the conditions of formation of the tetrachloride are obscure, and not less so is the cause of a phenomenon, noticed first by Claus, and since his day used as a test for the detection of ruthenium, and which is familiar to all of you who have experimented at all with this metal. I refer to the beautiful indigo-blue colour assumed by the solutions of ruthenium trichloride when hydrogen sulphide is led into them. Since, at the same time, sulphur is precipitated, and since the trichloride assumes a blue colour on treatment also with metallic zinc,* it was assumed by Claus that reduction has taken place, and hence that the solution contains ruthenium bichloride, $RuCl_4$. When ruthenium is heated in a current of mixed chlorine and carbon monoxide, it increases many times in volume, and there is formed an anhydrous trichloride. This is insoluble in water and in strong alcohol, but dissolves with considerable readiness in dilute alcohol to a similar deep blue solution. Joly succeeded in distilling off the alcohol and water from this solution, in a vacuum, and obtained a blue deliquescent substance which he considered to be an oxychloride, $RuOHCly$. I have also formed this blue solution by electrolytic action, and while it seems to be formed by a reducing action this is not perfectly clear.

Considerable work upon this solution, however, leads me to agree with Claus that it is probably a lower chloride of ruthenium, but it has not been proved. I have dwelt perhaps unduly upon these compounds, for the purpose of showing the obscurity in which even such seemingly simple points are enveloped, for it well illustrates how much work must yet be done before we acquire any adequate knowledge of the nature of even the commoner compounds and reactions of these elements.

(To be continued).

THE DISTRIBUTION OF MOLECULAR ENERGY.†

By J. H. JEANS, B.A.,
Scholar of Trinity College and Isaac Newton Student in the
University of Cambridge.

THIS Paper attempts to examine the well-known difficulties in connection with the partition of energy in the molecules of a gas. A definite dynamical system is first considered, an ideal gas in which the molecules are loaded spheres, that is spheres of radius a , of which the centre of mass is at a small distance, r , from the geometrical centre. It is shown by direct methods that the energy will, after an infinite time, distribute itself equally between the five degrees of freedom; but when a wave of sound is passed through the gas, the energy will never have sufficient time to attain to its equilibrium distribution. It is shown that sounds of different period will be propagated with appreciably different velocities, except in the extreme case in which the ratio of r to a is almost, but not necessarily quite, zero. In this case the ratio of the two specific heats, as determined from indirect experiments on the velocity of sound, would be $1\frac{1}{2}$, while direct experiments might give any value from $1\frac{1}{2}$ to $1\frac{1}{3}$, the value varying with the duration of the experiment.

It is suggested that an escape from this dilemma is made possible by regarding the molecules as forming an incomplete dynamical system, of which the ether is the remaining part. For purposes of illustration, it is imagined that the interaction between the two parts of this complete system consists of a frictional force which retards the rotation of the molecules. A steady state is

* It has already been mentioned that Vauquelin had noticed this blue colour, but not knowing of ruthenium had attributed it to osmium.

† Abstract of a Paper read before the Royal Society, June 21, 1900.

now impossible, but it is shown that when the energy (*i. e.*, temperature) of the gas is sufficiently low, the gas tends to assume an approximately steady state, in which the energy of rotation vanishes in comparison with that of translation.

It is then shown that these conclusions may be generalised so as to apply to a more complex system of molecules, these molecules possessing an indefinite number of degrees of freedom, and internal potential energy as well as kinetic. The molecules exert forces on one another at any distance, and the radiation is of a more general type than before.

In Part III. some of the physical consequences of the view here put forward are examined. The final conclusions are briefly as follows:—

The degrees of freedom must be weighted, not counted. The weight of a degree of freedom may be anything between unity and zero, and may vary with the temperature. A degree of freedom which does not radiate energy will always be of weight unity; for a non-luminous gas, one which does radiate energy when the gas is heated, is of weight zero.

As the gas is heated, the radiation and internal energies will increase much more rapidly than the temperature, until finally, at infinite temperature, the energy is distributed equally between all degrees of freedom.

Finally it is pointed out that this view is in accordance with ordinary thermodynamics for a non-luminous gas, but that the ordinary thermodynamics must be supposed to break down above the temperature of incandescence, a view which has already been put forward, in a modified form, by Wiedemann.

ANALYSIS OF THE NATURAL MINERAL WATER FROM THE No. 3 SPRING, BRAULT, SAIL-SOUS-COUZAN (LOIRE).

By EDMOND BONJEAN.

THIS water is particularly interesting by reason of the large amount of carbonic acid which it contains, and by its mineral richness, which causes it to be of the most mineralised type and of the greatest purity of any water in this hydro-mineral region.

The flow of this spring, measured by M. Coste, a well-known mining engineer, on February 17th, 1900, was 51 litres per minute: its invariable temperature is 12°.

The analyses were made on samples taken by me on August 27th, 1898.

The results are expressed in grms. per litre:—

I. Elementary Analysis.

	Grms.
Total carbonic acid	4.387
Combined carbonic acid	1.561
Free carbonic acid (1438 c.c.)	2.826
Silica, as SiO ₂	0.027
Oxide of iron, as Fe ₂ O ₃	0.011
Oxide of manganese	distinct traces
Lime, as CaO	0.126
Magnesia, as MgO	0.0997
Soda, as Na ₂ O	0.8205
Potash, as K ₂ O	0.1425
Lithia, as Li ₂ O	0.0025
Arsenic, as As	0.0005
Sulphuric acid, as SO ₃	0.0185
Chlorine, as Cl	0.0577
Ammonia and ammoniacal salts	traces
Organic matter	traces
Nitrates, nitrites, phosphoric acid, strontium	none
Residue at 110–120°	2.1305
Alkalinity in CO ₂ Na ₂	1.990

II. Combination of the Fixed Elements.

	Grms.
Silica, as SiO ₂	0.027
Ferrous carbonate, CO ₃ Fe	0.016
Carbonate of lime, CO ₃ Ca	0.225
" magnesia, CO ₃ Mg	0.2093
" lithia, CO ₃ Li	0.0062
" soda, CO ₃ Na	1.2918
" potash, CO ₃ K	0.2098
Sulphate of soda, SO ₄ Na ₂	0.033
Chloride of sodium, NaCl	0.095
Arsenate of sodium, AsO ₄ Na ₃	0.0014

2.1145

III. Probable Combination of the Elements in Solution.

	Grms.
Free carbonic acid (1438 c.c.)	2.826
Silica, as SiO ₂	0.027
Ferrous bicarbonate, FeCO ₃ .CO ₂	0.022
Bicarbonate of manganese	distinct traces
" lime, CaCO ₃ .CO ₂	0.3240
" magnesia, MgCO ₃ .CO ₂	0.3180
" soda, NaHCO ₃	2.0462
" potash, KHCO ₃	0.3032
" lithia, LiHCO ₃	0.0099
Sulphate of soda, SO ₄ Na ₂	0.0330
Chloride of sodium, NaCl	0.0950
Arsenate of sodium, AsO ₄ Na ₃	0.0014
Ammonia and ammoniacal salts	traces
Organic matter	traces

Total elements in solution 6.0057

Bacteriological Examination.—Cultivations conducted on the spot enable me to affirm that this water is sterile.—*Bull. Soc. Chim.*, Ser. 3, xxiii., No. 10.

ON ELECTRIC TOUCH AND THE MOLECULAR CHANGES PRODUCED IN MATTER BY ELECTRIC WAVES.*

By JAGADIS CHUNDER BOSE, M.A., D.Sc.,
Professor of Physical Science, Presidency College, Calcutta.

(*Notes added March, 1900.*—In the various investigations on the properties of electric waves, one property has not yet attracted so much attention as it deserves—the action of long ether waves in modifying the molecular structure of matter. Apart from the interest attached to the relation between ether, electricity, and matter, the subject is of importance as affording not only a very important verification of the identity of visible and electric radiation, but also establishing the continuity of all radiation phenomena. These phenomena occupy the borderland between physics and chemistry, and their study may therefore be expected to throw much light on several subjects at present imperfectly understood. The study of the action of electricity and of ether waves on matter in the form of solids presents many difficulties, owing to the great complexity of atomic and molecular aggregation which characterises the solid state of matter. But the phenomena often met with in theory and practice is, unfortunately, in reference to matter in a solid state. The means of investigation are almost wanting: chemical tests give us no information, for they tell us (and that in a few cases only) of the ultimate change in the mass as a whole, and not of the protean transformations that are constantly taking place in it under the action of ever-varying changes in physical environments. In the following investigations the difficulties mentioned above were constantly present, and the attempts to meet them may therefore be of some interest).

* A Paper read before the Royal Society, February 8, 1900.

In my former paper ("On a Self-recovering Coherer, and the Study of the Cohering Action of Different Metals," *Proc. Roy. Soc.*, vol. lxx.), I described the contact-sensitiveness of various elementary substances to electric radiation. It was shown that though many substances exhibit a diminution of contact-resistance, there are others which show an increase of resistance—an increase which, in certain cases, lasts only during the impact of electric waves, the sensitive substance automatically recovering its original conductivity on the cessation of radiation. There are thus produced two opposite effects, either an increase or a diminution of resistance, depending on the nature of the substance.

The effect of increase of contact-resistance is not an exceptional or isolated phenomenon, but is as normal and definite under varied conditions as the diminution of resistance noticed in the case of iron filings. These two specifically different effects have to be recognised, and it would be advisable, to avoid misunderstanding, to use a simple term to indicate both these effects, and distinguish them from one another, by calling the one positive and the other negative. The term "coherence" applied to the normal diminution of resistance exhibited by certain metals by the action of electric waves cannot be applied in all cases, as there is, as has been said before, another class of substances which exhibits under normal conditions an increase of resistance. The term "decoherence" has been used to indicate the effect of mechanical tapping on fatigued substances of the former class; this produces an increase of resistance, and at the same time restores the sensitiveness. The action of tapping on fatigued specimens of the latter class is, however, a diminution of resistance.

I have shown in a former paper that the seat of sensitiveness is confined mainly to the surface layer of the sensitive substance, and that the nature of the substratum has little or no effect on the sensitiveness. Thus, a substance which exhibits a strong diminution of resistance, if coated with an extremely thin layer of a substance of the other class which shows an increase of resistance, will now exhibit an increase of resistance. It is seen that the action is one of the bounding layer or skin of the sensitive substance. There is a Sanskrit word, *twach*, which means the skin; and as the phenomenon dealt with in the present paper is one of sensitiveness of *twach*, I shall use the expression "electric touch" in the restricted sense of contact sensitiveness to electric stimulus, the touch being regarded as *positive* when electric oscillation produces an increase of conductivity or diminution of resistance, and *negative* when the contrary effect is produced. Substances which exhibit a decrease of resistance will be called positive, and those which show an increase will be regarded as negative. The above terms are to be regarded as convenient substitutes for long descriptive phrases.

The phenomenon of contact-sensitiveness seems at first to be extremely anomalous, and there appears to be little relation between substances which exhibit similar electric sensitiveness. Taking iron as an example of a very sensitive substance, it is seen to be easily oxidised, and from this it may be inferred that a slight oxidation on the surface is favourable for sensitiveness. This view obtains some support from the consideration that the so-called noble metals are not as sensitive as iron. But the metals nickel and cobalt, which are bright and not easily oxidised, are also very sensitive. The very sensitive metals iron, nickel, and cobalt are all magnetic, and it might be thought that magnetic property is favourable for electric sensitiveness, but a dial magnetic substance like bismuth is also found to exhibit a fairly strong sensitiveness. Again, from the strong diminution of resistance exhibited by magnesium, it may be inferred that the sensitiveness depends on the electro-positive character of the metal; but potassium, one of the most electro-positive metals, exhibits an unusual increase of resistance, a property which it will be shown in a future paper it shares with some of the most electro-negative elements.

There is one property, however, which at first would seem to be in some way related to the sensitiveness of metals—the volatility of metals under the cathodic stimulus, investigated by Sir William Crookes (*Proc. Roy. Soc.*, vol. l.), who gives the following list of metals, arranged according to their volatility:—

Palladium	108
Gold	100
Silver	80.68
Lead	75.04
Tin	56.96
Brass	51.58
Platinum	44
Copper	40
Cadmium	31.99
Nickel	10.99
Indium	10.49
Iron	5.5
Magnesium	very
Aluminium	slight

In this list the substances which are most volatile, e.g., Pd, Au, Ag, are not very sensitive, whereas Fe, Al, Mg, which are least volatile, are strongly sensitive. But the above series does not exactly coincide with the series of electric sensitiveness. Again, the volatility of platinum is retarded in hydrogen gas, but an experiment carried out to determine the sensitiveness of platinum in hydrogen failed to show any great increase in the sensitiveness.

None of the above suppositions give any satisfactory explanation of the numerous anomalies in the contact-sensitiveness of metals. It then appeared that the observed effect is not due to a single cause but to many causes. An observer studying the dilatation of a gas under reduced pressure, and ignorant of the effect of temperature, will doubtless encounter many anomalies. In the phenomena of contact-sensitiveness the variables are, however, far more numerous, and the different possible combinations are practically unlimited. It therefore became necessary, by a long and tedious process of successive elimination, to find out the causes which are instrumental in producing the observed effect; the results obtained throw some light on this intricate subject. The following are some of the principal directions in which a systematic inquiry was carried out:—

- On the difference between mass action and molecular or atomic action, with reference to the phenomenon of contact-sensitiveness.
- On the change of sign of response in a receiver due to a variation of the radiation intensity.
- On the physico-chemical changes produced in a sensitive substance by the action of electric radiation, and on the radiation-product.
- The phenomena of electric reversal and of radio-molecular oscillation.
- On "fatigue" and the action of mechanical tapping and other disturbances by which the sensitiveness of a fatigued receiver may be restored.
- On the nature of the passage of electricity through imperfect contacts, and the influence of various physical agents on the current.
- On the systematic study of the contact-sensitiveness exhibited by metals, non-metals, and metalloids.
- On the contact-sensitiveness exhibited by alloys and compounds.

I intend to treat the above subjects in some detail, and in the present paper will especially deal with the first five lines of investigation. These, it is hoped, will afford an explanation of some of the most perplexing anomalies. All the subjects mentioned above are more or less interdependent, but their treatment in one paper would make the subject very complicated. It would be easier to take a more generalised and complete view of the subject as a whole, after each of the above-mentioned inquiries has been separately considered. With reference to the flow

of electricity through imperfect contacts, I need only mention here that the phenomenon seldom obeys Ohm's law. In many instances the phenomenon appears more allied to the discharge of electricity through a gaseous medium.

(To be continued).

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART III.

By HARRY BREARLEY.

(Concluded from p. 27).

CARBON (continued).

IV. COLORIMETRIC ESTIMATION OF CARBON.

IV.a. Eggertz Process and Modifications.—

429. EGGERTZ (C. N., xlv., 173).—Desirable relation between C in steel, HNO_3 , and state of dilution. Daylight bleaches colour. Comparison made in camera. Likely amounts of Mn, P, Si (see 444), S (see 434), W, Cr (see 435), Va, Ni, Co, and Cu are without influence. Permanent artificial standards. (See also 372).
430. HERMANN (C. N., xxi., 295).—Artificial standards not satisfactory. Process not suitable for high carbon steels.
431. GREINER (C. N., xl., 286).—The nitric solution of the steel is kept for four hours at 80° before comparing with the standard.
432. ROSSI (Y. I. S. I., 1891, i., 433).—Samples dissolved at 80 – 85° C., and the colours compared in a Duboscq colorimeter, which is described.
433. BRITTON (C. N., xxvi., 139).—Use 1 grm. of material; dissolve without heat, filter, and compare with series of standards (coffee in alcohol). Sample loses colour on standing.
434. HOGG (C. N., lviii., 175).—The S of a sample is liberated as such, and has a very marked influence on the tints.
435. ZIEGLER (Y. I. S. I., 1890, i., 373).—Chromium gives a grey tone to Eggertz colour solution.
436. TROILIUS (C. N., xlv., 292).—Compares between two standards not differing much in percentage from the sample.
437. SPÜLLER (Y. I. S. I., 1899, ii., 482).—After dissolving in HNO_3 , the samples are boiled in a paraffin bath (135° C.) before comparing.
438. STEAD (Y. I. S. I., 1883, 213; and C. N., xlvii., 285).—Dissolve 1 grm. in HNO_3 , add excess NaHO , and compare filtrate with similarly treated standard steel. Moderate excess of heating, HNO_3 and NaHO are without influence. HCl must be avoided. Hardened steels give less colour.
439. HUNT (Y. I. S. I., 1883, 764).—The mechanical state of division of standard and sample should be alike. Stead's modification the more reliable under variable conditions. The colour is darker the more quickly the sample is dissolved. Instructions for very quick testing.
440. PARKER (C. N., xlii., 88).—C exists in different conditions, so that steels containing same percentage give varying degrees of colour. The test is modified to meet this difficulty.
441. HOGG (C. N., xlii., 131).—Paper on same lines as Parker's. In hardened steel there is a form of C and iron "which is decomposed by HNO_3 without communicating any colour whatever."
442. HOGG (Y. I. S. I., 1896, ii., 179).—Classification of C existing in steel. Speculation concerning the carbon of hardened steel which evades the colour test. By continued heating, Eggertz colour is

destroyed and CO_2 evolved. Saniter connects the missing C with the sub-carbide Fe_{24}C , Sauveur with the presence of martensite.

443. GALBRAITH (Y. I. S. I., 1881, 234).—Hammering, rolling, hardening, and annealing interfere with the colour test. Conditions must be strictly comparative.
444. WOODCOCK (Y. I. S. I., 1882, 111).—Both P and Si introduce negative errors. Carbonaceous gases are given off on dissolving hardened steel.
445. PARRY and MORGAN (C. N., lxvii., 176).—Not reliable for hardened or high per cent steels. Artificial standards no use.
446. ROBINSON (Y. I. S. I., 1887, ii., 363).—Artificial standards made from CoCl_2 , CuCl_2 , Fe_2Cl_6 , and HCl .
447. UKENA (Y. I. S. I., 1891, ii., 323).—Samples cooled in hot soapy water can be drilled as though cooled in air.

IV.b. Tintometers and other Apparatus.—

448. LEEDS (C. N., xxxvii., 229).
449. STEAD (Y. I. S. I., 1883, 217; and C. N., xlvii., 285).
450. RIDSDALE (Y. S. C. I., 1886, 585).—With some notes on Stead's modified colour process.
451. RIDSDALE (Y. S. C. I., 1888, 70).—A simplified apparatus for deep tints.
452. LOVIBOND (Y. S. C. I., 1888, 424).
453. McMILLAN (Y. I. S. I., 1894, ii., 157).—Refers to previous instruments and describes two new ones.
454. KRÜSS (Y. C. S., lxvi., ii., 158).
455. ZANGEMEISTER (Y. C. S., lxx., ii., 404).
456. REDWOOD (Y. S. C. I., 1885, 76).
457. ARNOLD (C. N., i., 25).—Bath of glass; perforated lid of glazed earthenware. Tubes can be suspended any depth in the solution.
458. KELSALL (C. N., i., 56).—A copper bath. Burnt sugar standards keep for years.

V. OTHER PROCESSES OF ESTIMATING CARBON.

459. DUPRE (C. N., xxxix., 39).—Pass CO_2 into solution of basic lead acetate. The cloud formed is compared with standard clouds. Very delicate.
460. CLERC (Y. I. S. I., 1885, 274).—Liberate C with CuSO_4 , oxidise with CrO_3 , and pass CO_2 through tubes containing KHO and a little manganate. An excess of CO_2 forms permanganate; the number of tubes thus changed indicates the amount of C.
461. VOLMER (Y. I. S. I., 1895, ii., 587).—Reiper's process consists in making a metallic streak on unglazed porcelain, treating it with cuprammonium chloride, and comparing with standards.
462. TROPENAS and WELLS (Y. S. C. I., 1892, 636).—The CO_2 in converter gases is proportional to the C in the metal. This CO_2 is determined, and the C deduces in less than a minute. The process is patented.

VI. ESTIMATION OF GRAPHITE.

463. SHIMER (C. N., lxxiii., 31).—By decomposing with HCl or H_2SO_4 high results are obtained due to titanium and other carbides in the residue. These carbides are easily soluble in HNO_3 .
464. TOSH (C. N., xvi., 168).—Separate Fe with HCl , and wash residue with alcohol and ether to remove hydrocarbons. In separating Si with NaHO there is always effervescence due to oxidation to silicic acid.
465. EGGERTZ (C. N., xviii., 232).—Decompose with H_2SO_4 and HNO_3 , evaporate, and re-dissolve in HCl . Dry filtered graphite and SiO_2 on tared filter at 100° C. Loss on ignition = graphite.
466. PIESSE (C. N., xxix., 57).—Very much like 465. Dries at 212° .
467. ALLAN (C. N., xxix., 91).—Criticism of Piesse's paper. Dissolve in HCl . Silicon and carbon com-

- pounds removed with KHO, and graphite determined by loss on ignition of the strongly dried residue.
468. MACKINTOSH (C. N., li., 147).—To estimate graphite in minerals, fuse with KHO, wash residue with HCl, &c., dry, and weigh.
469. HOCK (Y. I. S. I., 1882, 324).—Ignite residue under charcoal cover in a tared crucible; weigh, ignite, and determine graphite by loss. Ignite the separated filter-paper, and from the residue calculated the graphite which adhered to the paper according to the ratio $\frac{\text{graphite}}{\text{ash}}$ established by the burning of the bulk of the graphite.
470. CROBAUGH (Y. I. S. I., 1894, li., 488).—Collect on counterpoised filters; remove SiO_2 with HF and HNO_3 ; digest with $(\text{NH}_4)\text{HO}$ to remove carbonaceous compounds.
471. TAMM (Y. I. S. I., 1887, li., 361).—Dries separated graphite at 100° , and divides residue, after ignition, by 0.94 to allow for water in SiO_2 .
472. PARRY (C. N., lxvii., 247).—Separate Fe with HCl, wash residue with KHO, alcohol, and ether; mix with CuO and burn in O, or dry on weighed paper at $100\text{--}120^\circ\text{C}$.
473. STOLBA (Y. S. C. I., 1888, 235).—The combustion of graphite is promoted by mixing with silver dust.
474. WIDMER (Y. C. S., lvi., 923).—Cross and Bevan's observation that with CrO_2 part of the C is gasified as CO is true for graphite. To avoid error (5 per cent) the gases are passed over CuO.
475. SHIMER (C. N., lv., 231).—To sample graphitic irons moisten borings with alcohol, remove a portion, dry and weigh.* (See also Y. I. S. I., 1889, li., 472).

VII. MISCELLANEOUS NOTES.

476. WARREN (C. N., xlv., 85).—An interesting account of the combustion of organic bodies in oxygen.
477. BERTHELOT (C. N., xix., 113).—"Immediate Analysis of Different Varieties of Carbon."
478. LEDEBUR (C. N., xlvii., 107).—To remove grease from the sample ignite in a current of dry nitrogen.
479. RAWSON (C. N., xlix., 161).—"The Estimation of Cuprous Chloride in Copper Liquors."
480. DONALD (C. N., lxiii., 73).—By rusting, pig-iron drillings lose a portion of their graphite.
481. LEEDS (Y. I. S. I., 1881, 658).—"Burnt" steel contains the same amount of C as the same steel before burning.
482. HEMPEL (C. N., lix., 277).—Processes for estimating C classified and criticised. Ullgren (409), Weyl (334), and Wöhler's (378) processes preferred.
483. LEDEBUR (Y. I. S. I., 1894, i., 603).—Direct combustion of grey pig gives low results. Juptner's method (319) gives low results, due to escape of hydrocarbons. With Sarnström's modification (414) the results are accurate. Similar remarks apply to wet combustion of the C residue. Removal of Fe by cuprammonium chloride unsatisfactory. Separation of Fe with Cl most accurate if well done. HNO_3 preferable to HCl for separating Fe and graphite.
484. GÖTTIG (Y. I. S. I., 1894, i., 607).—Dry direct combustion inaccurate. Juptner's process (319) available if much CrO_2 is used. Cu and C need not be separated for wet combustion; must be for dry. Processes separating the iron and burning the residue in the dry way are inaccurate.
485. DOUGHERTY (Y. I. S. I., 1897, i., 573).—In FeSi compounds determines Fe, Si, Mn, S, and P, and then calculate the carbon by difference.

* Converted bar should be sampled by boring clean through the bar and using all the borings. Steel faced articles, such as "draw plates," may be examined by taking a broad thin cut from each face and a small hole quite through. The data from the three samples enables one to calculate the depth of steel facing.

486. TABARY (Y. I. S. I., 1895, i., 432).—Irregular distribution of carbon in pig-iron.
487. LUZI (Y. I. S. I., 1893, li., 334).—Graphite, so-called, may contain "graphitic" also. They behave differently with nitric acid.
488. LEDEBUR (Y. I. S. I., 1889, i., 368; and 1893, li., 53).—Distinguishes hardening C, carbide C, graphitic temper C, and graphite; their properties and means of estimating them.
489. LEDEBUR (Y. I. S. I., 1891, i., 364).—Analysis of variously treated iron and steels, including graphite, graphitic temper carbon, hardening carbon, and carbide carbon.
490. JUPTNER (Y. I. S. I., 1897, i., 248).—Estimates hardening and carbide C by a modification of the colour test. Summary of Osmond and Werth's investigation of the colour test. Graphite is oxidised on boiling with HNO_3 . Bamber (p. 266) vigorously criticises the methods. (See also p. 554).
491. DIXON (Y. C. S., lix., 774).—"Mode of Formation of CO_2 in the Burning of Carbon Compounds."
492. ABEL and DERRING (C. N., xlvii., 200).—Carbon exists in cold rolled steel as Fe_3C . It is separated with a dilute solution of CrO_3 and H_2SO_4 . See also MULLER (Y. I. S. I., 1895, i., 495), ARNOLD and READ (Y. C. S., lix., 788), and MYLIUS, FOERSTER, and SCHOENE (Y. C. S., lixii., li., 39).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

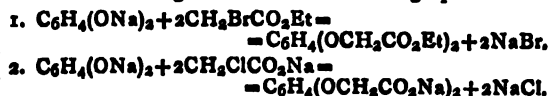
Ordinary Meeting, June 21st, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

(Concluded from p. 34).

98. "The Oxyphenoxy- and Phenyleneoxy-acetic Acids." By W. CARTER and W. TREVOR LAWRENCE.

The phenyleneoxyacetic acids, $\text{C}_6\text{H}_4(\text{OCH}_2\text{CO}_2\text{H})_2$, are obtained according to either of the following equations:—

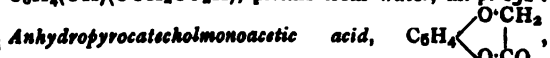


The condensation according to the first equation is not complete in the cases of resorcinol and hydroquinol, and a certain amount of acid ester, $\text{C}_6\text{H}_4(\text{OH})(\text{OCH}_2\text{CO}_2\text{Et})$, corresponding to the oxyphenoxyacetic acids, is formed, and may be separated from the neutral ester by potash solution.

On pouring the esters into alcoholic potash the potassium salts of the acids are precipitated as microcrystalline powders, which when acidified yield the free acids.

The amides are obtained from the esters by shaking with aqueous ammonia, the aniline salts result on boiling the acids with a benzene solution of aniline, and the anilides are obtained by heating the acids to 190° with aniline.

Pyrocatechol Derivatives.—Ethyl pyrocatecholdiacetate, $\text{C}_6\text{H}_2(\text{OCH}_2\text{CO}_2\text{Et})_2$, boils at $230\text{--}232^\circ$ (30 m.m.). Pyrocatecholdiacetic acid, $\text{C}_6\text{H}_2(\text{OCH}_2\text{CO}_2\text{H})_2$, needles from water, m. p. 178° . Aniline salt, m. p. 250° . Barium salt, $2(\text{C}_{10}\text{H}_8\text{O}_6\text{Ba})\cdot\text{H}_2\text{O}$. Pyrocatecholdiacetanilide, $\text{C}_6\text{H}_2(\text{OCH}_2\text{CONHPh})_2$, m. p. 196° . Pyrocatecholdiacetamide, $\text{C}_6\text{H}_2(\text{OCH}_2\text{CONH}_2)_2$, m. p. 203° . Ethyl pyrocatecholmonoacetate, $\text{C}_6\text{H}_3(\text{OH})(\text{OCH}_2\text{CO}_2\text{Et})$, b. p. 155° (30 m.m.). Pyrocatecholmonoacetic acid, $\text{C}_6\text{H}_3(\text{OH})(\text{OCH}_2\text{CO}_2\text{H})$, prisms from water, m. p. 152° .



prisms from ligroin, m. p. 52°. *Pyrocatecholmonoacetanilide*, $C_6H_4(OH)(OCH_2CONHPh)$, m. p. 161°. (Compare Moureu, *Bull.*, 1899, [3], xxi., 107).

Resorcinol Derivatives.—*Ethyl resorcindiacetate*, $C_6H_4(OCH_2CO_2Et)_2$, m. p. 42° (B. 40°), b. p. 228° (32 m.m.). *Resorcindiacetic acid*, $C_6H_4(OCH_2CO_2H)_2$, m. p. 195°. The silver, copper, and iron salts are amorphous, the calcium and barium salts crystalline. *Aniline salt*, m. p. 137°. *Resorcindiacetanilide*, $C_6H_4(OCH_2CONHPh)_2$, m. p. 169° (B. ca. 182°). *Imidoester*,—



m. p. 43°. *Resorcindiacetamide*, $C_6H_4(OCH_2CONH_2)_2$, m. p. 167°. 2 : 4 : 6-*Trinitroresorcindiacetic acid*, $C_6H(NO_2)_3(OCH_2CO_2H)_2$, is formed by boiling resorcindiacetic acid with nitric acid, m. p. 174°, converted by potash at 140° into styphnic acid. *Resorcinmonoacetic acid*, $3(C_6H_5O_4)_2 \cdot H_2O$, m. p. 158°, anhydrous needles from toluene, m. p. 160° (B. 158°). *Resorcinmonoacetanilide*, m. p. 125°.

Hydroquinol Derivatives.—*Ethyl hydroquinoldiacetate*, $C_6H_4(OCH_2CO_2Et)_2$, needles from ligroin, m. p. 72°. *Hydroquinoldiacetic acid*, $C_6H_4(OCH_2CO_2H)_2$, prisms insoluble in all solvents tried except acetic acid, m. p. 251° (B. ca. 246°). *Hydroquinoldiacetanilide*, $C_6H_4(OCH_2CONHPh)_2$, m. p. 210°. The ammonium salt crystallises in needles from water. The silver and copper salts are amorphous, and the calcium and barium salts crystalline.

Hydroquinolmonoacetic acid, $3(C_6H_5O_4)_2 \cdot H_2O$, prisms from water, anhydrous needles from toluene, m. p. 152°. *Aniline salt*, m. p. 119°. *Hydroquinolmonoacetanilide*, $C_6H_4(OH)(OCH_2CONHPh)$, m. p. 101°.

The melting-points in brackets, and indicated by B, refer to a private communication from Prof. C. A. Bischoff, who is engaged in the investigation of many of these substances, in pursuance of his "Studies on Condensations," and to whom we therefore relinquish their further study.

99. "The Condensation of Ethyl α -Bromoisobutyrate with Ethyl Malonates and Ethyl Cyanacetates: α -Methyl α' -Isobutylglutaric Acid." By W. TREVOR LAWRENCE, B.A., Ph.D.

The author showed that the following general reactions hold good:—



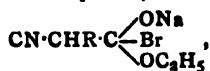
when the sodium compound is in suspension,



when the sodium compound is in solution, where R = H or an alkyl group; X = (CN) or (CO₂Et).

The behaviour of ethyl bromoisobutyrate is probably influenced by the degree of ionisation of the sodium compound.

The author suggests that the hydrobromic acid liberated from the bromoisobutyric ester combines with (the enolic form of) ethyl sodiomalonate or -cyanacetate to form a hypothetical addition compound; for example,—



which condenses with the unsaturated ester with separation of sodium bromide.

The following substances were isolated and investigated in the course of this research:—

I. In the condensation of ethyl sodioisobutylmalonate with ethyl α -bromoisobutyrate, or ethyl α -methacrylate in alcoholic solution.

Ethyl α -methyl- α' -isobutylpropanetricarboxylate, $CH(CH_3)CO_2Et \cdot CH_2 \cdot C(CH_3)(CO_2Et)_2$, b. p. 185° (18 m.m.), converted by hydrolysis with alcoholic potash into the potassium salt of α -methyl- α' -isobutylpropanetricarb-

oxylic acid. This acid melts at 167–168° (with evolution of carbon dioxide and formation of *cis*- and *trans*-methylisobutylglutaric acids).

The *cis*- and *trans*-methylisobutylglutaric acids, $CH(CH_3)(CO_2H) \cdot CH_2 \cdot CH(C_4H_9)CO_2H$, are best separated by crystallisation from boiling ligroin (b. p. 110–120°).

cis-Acid, m. p. 121°. *Anhydride*, a liquid, b. p. 196° (50 m.m.). *Anilic acid*, $C_{16}H_{23}O_3N$, m. p. 164°.

trans-Acid, m. p. 86–87°. *Anhydride*, a liquid, b. p. 178° (22 m.m.). *Anilic acid*, m. p. 196°.

The *trans*-acid may be converted into the *cis*-modification by heating with hydrochloric acid in a sealed tube, or into the *cis*-anhydride by distillation or by heating with acetic anhydride to 220°.

II. In the condensation of ethyl sodioisobutylcyanacetate, b. p. 165° (50 m.m.), with ethyl bromoisobutyrate or ethyl α -methacrylate in alcoholic solution; in the condensation of ethyl sodiocyanacetate with ethyl methacrylate and subsequent addition of isobutyl bromide.

Ethyl α -methyl- α' -isobutyl- α' -cyanoglutarate, $CH(CH_3)CO_2Et \cdot CH_2 \cdot C(CH_3)(CN)(CO_2Et)$, b. p. 196° (25 m.m.), on hydrolysis with alcoholic potash is converted into the potassium salt of a substance, $C_{11}H_{17}O_4N$, which melts at 164°, with evolution of carbon dioxide. This substance is possibly a mixture of methylisobutylcyanoglutaric acid with the monocarboxylic acid of methylisobutylglutarimide, as on distillation (295°, 760 m.m.) the imide (m. p. 78°) of the *cis*-acid (insoluble in sodium carbonate) and the nitrile of the *trans*-acid are obtained.

It is converted by hydrochloric acid into a mixture of *trans*-glutaric acid and *cis*-glutarimide.

The complete hydrolysis of the nitrile or imide is effected by 50 per cent sulphuric acid.

III. In the condensation of ethyl bromoisobutyrate with ethyl sodioisobutylmalonate or -cyanacetate in benzene solution; in the condensation of ethyl bromoisobutyrate with ethyl sodiocyanacetate in alcoholic solution with subsequent addition of isobutyl bromide.

Ethyl α -dimethyl- α' -isobutyl- α' -cyanosuccinate, b. p. 180° (20 m.m.), $CO_2Et \cdot C(CH_3)_2 \cdot CH(C_4H_9)(CN)(CO_2Et)$, when hydrolysed with alcoholic potash and then acidified, gives a pasty mass of cyano-acid—on complete hydrolysis with 50 per cent sulphuric acid it gives α -dimethyl- α' -isobutylsuccinic acid, $CO_2H \cdot C(CH_3)_2 \cdot CH(C_4H_9)CO_2H$, prisms from water, m. p. 141°.

Experiments on the oxidation of methylisobutylglutaric acid (the study of which was the original object of this research) with potassium permanganate either at 60° or in the cold, showed that the greater part of the acid could be recovered unchanged, the oxidation products consisting of oxalic acid, isobutylmalonic acid, a fatty acid (isobutylacetic acid?), two liquid acids, of which one appeared to be a hydroxy-acid, $C_{10}H_{18}O_5$, and the other an unsaturated acid, $C_{10}H_{16}O_4$, and a crystalline acid, m. p. 80°, of the same composition.

These acids were obtained in very small quantities and their complete separation was both difficult and probably unsuccessful.

100. "Methylisoamylsuccinic Acid." II. By W. TREVOR LAWRENCE, B.A., Ph.D.

The anil of *trans*- α -methyl- α' -isoamylsuccinic acid melts at 118°, and that of the *cis*-acid at 116°.

The partial conversion of the *cis*- into the *trans*-modification may be brought about by heating with hydrochloric acid, and the reverse change by heating the *trans*-acid with acetic anhydride at 210°.

The *cis*-anhydride is converted by phosphorus pentabromide, bromine, and alcohol into ethyl α -bromo-methylisoamylsuccinate (b. p. 155°, 20 m.m.), $C_{14}H_{25}O_4Br$, which, when poured into hot alcoholic potash, forms the potassium salt of isoamylcitraconic acid, $CO_2H \cdot C(CH_3) \cdot C(C_5H_{11})CO_2H$.

Oxidation of methylisoamylsuccinic acid by means of

potassium permanganate gave results similar to those obtained by the oxidation of the isomeric methylisobutylglutaric acid (compare preceding abstract); only a small portion of the acid was oxidised by the permanganate, giving oxalic acid, a liquid acid having the formula $C_{10}H_{16}O_4$, and a lactic acid having the same formula and melting at 103° .

Correction.—Isomylsuccinic acid melts at $83-84^\circ$, not 76° as previously stated (*Proc.*, 1899, xv., 163).

101. "The Estimation of Furfural." By WILLIAM CORMACK.

The process proposed by the author is based on the oxidation of furfural to pyromucic acid by means of an ammoniacal solution of silver oxide according to the equation $C_5H_4O_2 + Ag_2O = C_5H_4O_3 + 2Ag$. The action takes place quantitatively when the solutions are warmed. A known volume of a decinormal silver oxide solution, somewhat in excess of that required for the oxidation of the furfural, is added to the furfural solution, the reduced silver is filtered off through asbestos, and the silver remaining in the filtrate estimated by means of decinormal ammonium thiocyanate.

The method is applicable to furfural solutions such as those derived from fibres by distillation with hydrochloric acid, the furfural being distilled over with steam from the solution after neutralisation with alkaline carbonate, and subsequent slight acidification with oxalic acid.

102. "The Constitution of Hydrogen Cyanide." By JOHN WADE.

When potassium cyanide is heated with alkyl potassium sulphates at a lower temperature than in the preparation of nitriles, the isomeric isocyanide is often the principal product. The author also has found that practically all the isocyanides can be converted into nitriles by the action of heat. The formation of nitriles in the above interaction is thus accounted for, and one of the arguments in favour of the nitrilic constitution of hydrogen cyanide disappears.

Isocyanides form crystalline products with aldehydes, probably homologous with the aldehyde cyanhydrins, for which the formula—



is suggested, as the ordinary formula does not account for their anomalous behaviour with alkalis. Compounds have in this way been obtained from acetaldehyde and benzaldehyde in conjunction with methyl-, ethyl-, and phenyl-isocyanides.

Isocyanides also form crystalline products with alkyl iodides in presence of alcohol. This interaction accounts for the non appearance of isocyanides, and probably for the formation of nitriles, when potassium cyanide is heated with alkyl iodides in presence of alcohol. Compounds of this class have been obtained from methyl-, ethyl-, propyl-, isopropyl-, butyl-, isoamyl-, and phenyl-isocyanides in conjunction with methyl, ethyl, propyl, isopropyl, isobutyl, and isoamyl iodides.

n-Propyl isocyanide, and *n*-butyl isocyanide do not appear to have been prepared before. They are formed by interaction of the alkyl iodides with silver cyanide, and boil respectively at $97-99^\circ$ and $118-120^\circ$ (uncorr.).

103. "Inhibiting Effect of Etherification on Substitution in Phenols." By HENRY E. ARMSTRONG and EDWARD W. LEWIS.

The inhibiting effect on substitution produced by introducing methyl, ethyl, or benzyl in place of the hydroxylic hydrogen in phenolparasulphonic acid has already been referred to (*Armstrong, Proc.*, 1899, xv., 177). At the last meeting of the British Association, it was pointed out that "benzoyl appears to exercise a very remarkable inhibitive effect, as preliminary experiments show that benzoylated phenolparasulphonic acid remains unattacked by bromine under conditions which involve the conversion of the unbenzoylated acid into tribromophenol." This observation

has since been confirmed, and the experiments have been extended in order to determine the effect of radicles generally. Besides benzoyl, phenylsulphonyl, $Ph \cdot SO_2 \cdot$, benzylsulphonyl, $Ph \cdot CH_2 \cdot SO_2 \cdot$, and the radicle of Reychler's camphorsulphonic acid, $C_{10}H_{15}O \cdot SO_2 \cdot$, afford complete protection against bromine. Picryl, $C_6H_2(NO_2)_3 \cdot$, also appears to behave like benzoyl, but owing to the instability of picrylphenolparasulphonate in aqueous solution it is difficult to determine the exact nature of the change effected by bromine. Acetyl, on the other hand, exercises an effect similar to that produced by methyl and ethyl; and the complex radicle phenacyl, $Ph \cdot CO \cdot CH_2 \cdot$, derived from acetophenone, acts much in the same way as acetyl. Difficulty has been experienced in determining the influence of the isomeric radicle phenacetyl, $Ph \cdot CH_2 \cdot CO \cdot$, owing to the instability of the ether produced by it; apparently, it also resembles acetyl.

In order to ascertain whether a determining influence is exercised by hydrogen in association with the carbon atom which becomes attached to the phenolic oxygen, tertiary butyl was introduced into phenolparasulphonate. The effect produced by this radicle seems to be altogether different from that of either methyl or ethyl, because on treating the sulphonate with one molecular proportion of bromine, only monobromosulphonate is obtained, the sulphonic radicle remaining intact, but it is displaced by a second molecular proportion of bromine; the methyl and ethyl derivatives are, to a large extent, directly converted into the parabromophenol ethers. In view of this observation, it will be necessary to examine other alkyl derivatives.

104. "Bromination of Oxyazo-compounds." By HENRY E. ARMSTRONG and PERCY C. C. ISHERWOOD.

The observations recently made by Hewitt and Aston and by Auwers unquestionably prove that the compound formed by the interaction of phenol and a diazobenzene salt, behaves, in the main, as benzeneazophenol, and there is no doubt that the isodynamic hydrazone comes prominently into evidence only in the presence of acids; it is clear, however, that the equilibrium is easily disturbed in either direction, and that in such a case the constitution can only be finally inferred from physical rather than chemical properties.

The authors must confess to having been too much influenced by the extraordinary difference in the behaviour of this compound towards excess of bromine as compared with that of its ethylated derivative, but apparently it is not necessary to infer from this that the two are structurally different, as the principle developed by one of them in discussing the laws which govern substitution in benzenoid compounds suffice to afford an explanation of the dissimilarity.

The conversion of benzeneazophenol into benzeneazodibromophenol and its re-solution by excess of bromine into diazobenzene and tribromophenol are comparable with the conversion of phenolparasulphonic acid, first into the dibromosulphonate, and then into tribromophenol and sulphuric acid.

Ethylation exercises an inhibiting influence such as is referred to in the previous abstract, benzeneazophenol being converted by bromine in presence of sodium acetate only into benzeneazomonobromophenol. This compound is also formed when a solution of the phenol in glacial acetic acid is brominated, but at the same time a "hydrobromide perbromide" is formed which is readily deprived of its bromine by reducing agents. In a previous note (*Proc.*, 1899, xv., 243), the mistake was made of inferring from observations made with this latter compound that benzeneazo-ortho-bromophenol is an easily reducible substance.

As the separation of the azo-group from Hewitt and Aston's benzeneazodibromophenol by bromine is effected even in presence of sodium acetate, it cannot be argued that the scission is consequent on the formation of the hydrazone, and the stability of benzeneazophenol

must therefore be ascribed to the change produced by displacing the hydroxylic hydrogen. The extreme readiness with which the compound assumes the hydrazone form is manifest from the fact that if the sodium acetate be omitted, and two molecular proportions of bromine are used, the substance being merely dissolved in acetic acid, benzeneazodibromophenol is mainly resolved into bromodiazobenzene and tribromophenol. Benzoylated benzeneazophenol, like benzoylated phenolparasulphonic acid, is unaffected by bromine.

Clearly it will be desirable to study further oxy- as well as amido-azo compounds from the points of view indicated in the preceding abstract.

105. "*Meta-sulphonation of Aniline.*" By HENRY E. ARMSTRONG and W. BERRY.

It has been pointed out by one of the authors that, "in order to produce meta-derivatives from amines, it is necessary to paralyse, as far as possible, the ordinary ortho-para-orientating influence of the amino-group, and to give opportunity for the attack to take place in the nucleus" (*Proc.*, 1899, xv., 177; compare *B. A. Report*, 1899, 685, § 11).

In the case of aniline, sulphuric acid alone appears to be capable of exercising the necessary protective influence, because if the sulphate be dissolved in a large excess of chlorosulphonic acid, the mixture may be heated to 30–40° C. without any appreciable amount of hydrogen chloride being evolved. Under these conditions, the chlorosulphonic acid is not a sufficiently powerful sulphonating agent to produce even the sulphamate. Consequently, if aniline sulphate be added to a sufficiently strong fuming sulphuric acid, it is in part converted into the meta-sulphonic acid, the nucleus apparently being directly attacked. It is therefore possible to convert aniline into either para- or ortho- or meta-sulphonic acid at will.

106. "*Phenylacetylchloramine and Analogous Compounds.*" By HENRY E. ARMSTRONG.

The author calls attention to the discrepancies in the descriptions given of the properties of phenylacetylchloramine, and discusses the manner in which it under goes isomeric change.

107. "*Benzylaminesulphonic Acids.*" By IDA SMEDLEY. These acids have been prepared in order to compare their behaviour with that of the corresponding methyl and ethyl derivatives studied by Miss Evans (*Proc.*, 1895, xi., 235; 1896, xii., 234).

Anilineparasulphonic acid is readily benzylated by digesting an aqueous solution of its sodium salt with benzyl chloride and alkali, the mono- or di-benzyl derivative being formed according to the proportions used. The meta-acid is dibenzylated with extreme readiness.

In their behaviour with bromine, the dibenzylated closely resemble the dimethylated acids. It may therefore be assumed that benzyl does not promote the displacement of the sulphonic group as it does when introduced into phenolparasulphonic acid in place of the hydroxylic hydrogen, but it undoubtedly has an influence different from that exercised by either methyl or ethyl. Thus it is characteristic of dimethyl and diethylsulphanilic acids that, when acted on by bromine, each is converted into an ortho-brominated acid capable of combining with bromine to form a comparatively stable perbromide. The dibenzyl acid yields a similar monobrominated acid, but this does not form a perbromide; it is, however, very easily deprived of its benzyl by the further action of bromine.

108. "*Benzeneorthodisulphonic Acid.*" By HENRY E. ARMSTRONG and S. S. NAPPER.

The authors have prepared benzeneorthodisulphonic acid by applying Leuckart's xanthate method (*J. pr. Chem.*, 1890, xli., 179, to parabromanilineorthosulphonic acid. A series of compounds has thus been obtained corresponding with those prepared by Miss Walter (*Proc.*, 1895, xi., 141) from sulphanilic acid.

Potassium parabromophenylxanthateorthosulphonate is easily soluble in water, from which it crystallises with 10 molecules of water.

When hydrolysed by means of sulphuric acid or alcoholic potash, it yields the corresponding thiophenolsulphonic acid, $C_6H_4Br(SH) \cdot SO_3H$, or its potassium salt, the thiophenetsulphonate, $C_6H_4Br(SEt) \cdot SO_3K$ being also formed when it is hydrolysed by aqueous potash, and when it is decomposed by heating at about 200°.

Bromobenzene-3:4-disulphonic acid is conveniently prepared by directly oxidising the xanthate with potassium permanganate. Its sulphochloride crystallises in monosymmetric prisms melting at 88°. The amide crystallises in brilliant anorthic prisms; the anilide in monosymmetric plates melting at 182°. It is converted into benzeneorthodisulphonic acid by boiling its aqueous solution with soda and zinc dust.

Sodium benzeneorthodisulphonate is very soluble in water, from which it crystallises in long transparent prisms. The barium salt is sparingly soluble even in hot water, and crystallises in glistening plates.

Unlike 1:2-naphthalene-1:2-orthodisulphonic acid, but like toluene 3:4-disulphonic acid, benzeneorthodisulphonic acid is converted into the corresponding chloride by phosphorus pentachloride. This crystallises in magnificent monosymmetric prisms melting at 143°.

The corresponding amide and anilide also crystallise in monosymmetric prisms, the former melting at 254°, the latter at 241°.

A comparison of the acid with phthalic acid is being made.

109. "*An Isomeride of Furfurine.*" By J. P. MILLINGTON, B.Sc., and H. HIBBERT, B.Sc.

By the application of the method of Japp and Moir (*Trans.*, 1900, lxxvii., 637) for the preparation of isomarine from amarine, the authors have succeeded in preparing an isomeride of furfurine. It crystallises from water in needles melting sharply at 143° (furfurine melts at 116°), and on analysis was found to correspond with the formula $C_{13}H_{12}N_2O_5$. A platinumchloride and silver derivative have been obtained of which the formulae are $(C_{13}H_{12}N_2O_5)_2H_2PtCl_6$ and $C_{13}H_{11}N_2O_5Ag$ respectively.

110. "*The Mono- and Di-acetyl and Phenacetyl Diethyl Tartrates.*" By J. MCCRAE and T. S. PATTERSON.

The preparation of monoacetyl, diacetyl, monophenacetyl, and diphenacetyl diethyl tartrates was described. Considerable difficulty was experienced in the purification, and different methods had to be adopted in each case. The specific rotation of these substances has been determined at various temperatures with the following results:

Monoacetyl diethyl tartrate	..	$[\alpha]_D^{20} = +9.30^\circ$
Diacetyl	..	$[\alpha]_D^{100} = +6.30^\circ$
Monophenacetyl	..	$[\alpha]_D^{20} = +30.38^\circ$
Diphenacetyl	..	$[\alpha]_D^{20} = +17.92^\circ$

A comparison is instituted, on the one hand, between the series of monoacidyl tartrates, where it is shown that the phenacetyl radicle exerts a much greater influence than that exerted by the acetyl radicle, and, on the other hand, between the members of the series of diacidyl tartrates where the acetyl and phenacetyl radicles behave similarly but very differently from the toluyl radicles.

The regularity of the influence exerted by the successive introduction of two acidyl groups is noticed. The first acidyl group increases the rotation of the diethyl tartrate, but the second acidyl group diminishes the rotation of the monoacidyl compound.

Development and Propagation of the Explosive Wave.—H. Le Chatelier.—The author's experiments were performed on mixtures of acetylene with oxygen and the oxygen compounds of nitrogen, and on an explosive mixture of carbonic oxide and oxygen.—*Comptes Rendus*, cxxx., No. 26.

CORRESPONDENCE.

DISTILLATION WITHOUT BUMPING.

To the Editor of the Chemical News.

SIR,—In view of the number of twice-told tales that are continually finding their way into the literature of chemistry and other sciences it is doubtless time to make a glaring example of somebody, but in view of the numerous *able-bodied* Hawthornes in chemistry it does seem almost cruel, not to say cowardly, to select for this purpose the writer of the poor pitiful little pipe-stem squib that appeared in this journal under the above title, and we venture to predict that if the ghost who stuck the match to this victim would but dare to publish his name so that his writings could be reviewed from the same standpoint, he would thereafter do his haunting on some other planet.—I am, &c.,

DIAZO.

NOTICES OF BOOKS.

The Bacterial Treatment of Sewage; a Handbook for Councillors, Engineers, and Surveyors. By GEORGE THUDICHUM, F.C.S., &c. London: The Councillor and Guardian Offices. Pp. 92.

HITHERTO chemical treatment of some sort has occupied a prominent place in all books dealing with the subject of the purification of sewage. These methods of precipitation, sludge treatment, &c., are, according to our author, no longer considered, though the knowledge acquired during the past few years may cause the profitable utilisation of waste matters to be to some extent attainable.

Experience has shown that the best methods of sewage treatment depend on bacterial action in specially prepared areas. And, in the author's opinion, there are only two adaptations of this principle which have been proved of practical value. These two methods are the Sutton system and the septic tank, by either of which a constantly good result can be obtained in the simplest and cheapest possible manner.

The Sutton or aerobic system of sewage purification is the outcome of the experiments carried out by the Metropolitan Board of Works and the London County Council, between the years 1884 and 1896. In this method the sewage first passes through a grid, and then into a coarse-grained bacteria bed consisting of a tank filled with lumps of coke, broken bricks, clinker, &c., which will pass through a three inch ring, but no fine stuff; it is under-drained with open jointed drain-pipes leading to the discharge point. After filling with sewage the bed is allowed to stand for two hours; it is then emptied on to another finer-grained bed, and left for a similar time. In these beds the organic matter in solution is oxidised, and the suspended matter deposited; after discharging, the sludge remains in the bed, the interstices of which now being filled with air, the aerobic organisms are provided with all the conditions for rapid reproduction, the organic matters are rapidly attacked, and after a few hours the bed is ready for another charge. Practical experience has shown that these beds may work twice daily with advantage. The septic tank process devised by Mr. Donald Cameron differs principally from the Sutton system in that in the former case anaerobes, and in the latter case aerobes, are chiefly relied on to effect the solution of the suspended matter, and the preliminary breaking down of complex organic substances. The tank is made of such a size that the sewage takes from eighteen to twenty-four hours to pass through, and during this time the organic matter in suspension is dis-

solved by bacterial action in such a manner that the effluent leaving the tank is oxidised to the extent of some 50 per cent, while the whole is in a condition to be readily purified in the oxidising aerobic beds; these beds, by an ingenious device, have the filling, standing full, emptying, and resting empty for aeration regulated automatically.

Amongst other methods described by the author we may mention those of Scott, Moncrieff, Lowcock, Adeney, Waring, &c.; they all, however, are said to be inferior to the Sutton and septic tank systems, without producing better results.

The author also deals with the treatment of storm water, trade refuse liquors, and the land treatment of sewage, but the great merit of his book lies in the fact that it is the first book which deals solely with the modern bacteriological methods of sewage purification.

Elementary Inorganic Chemistry. Metals. By S. R. TROTMAN, M.A., F.I.C., &c. London: Rivingtons. 1900. Pp. 182.

THIS volume is intended as a continuation of the author's book on the non-metallic elements, and in a like manner it gives a selection of the more important facts concerning the elements dealt with, in a style suitable for a first course.

The metals and their principal reactions are first described separately; then in Chapters XI. to XV. the group arrangements are given with tables for making simple analyses.

The book is well arranged and clearly printed, and will make a useful laboratory companion for beginners.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxx., No. 26, June 25, 1900.

The Acidity of Alcohols.—M. de Forcrand.—The author recently determined the following influence coefficients for carbon and hydrogen:—

$$C = +3.01 \quad H = -2.85;$$

and from these obtained values for—



By means of these and other known numbers the following coefficients were obtained:—

Monatomic alcohols	..	+29.71 to 27.89	(from C to C ₃).
Glycol		+31.32	"
Glycerine		+32.28	"
Erythrite		+32.76	"

Hydrogenation of Ethylene in presence of various Reducing Metals.—Paul Sabatier and J. B. Senderens.—The series of experiments performed by the authors show that, in the same manner as reduced nickel, so reduced cobalt, copper, and iron are capable of inducing the hydrogenation of acetylene, but their activity is less. Whilst nickel reacts in the cold and indefinitely, cobalt only acts in the cold for a very limited time: when hot its action is tolerably rapid, but diminishes in proportion to the carburization of the metal. Copper acts still less quickly and only above 180°, but the action does not diminish, and can, under these conditions, effect the total hydrogenation after a considerable lapse of time. Iron is the slowest of all; above 180° even it acts slowly, and

its activity, very slight in the beginning, diminishes little by little.

Crystalline Compounds of Acetylene with Cuprous Chloride and Potassium Chloride.—M. Chavastelon.—In a previous paper the author showed how, by regulating the current of acetylene through a solution of cuprous chloride and potassium chloride, either yellow or colourless crystals can be obtained. He showed that it was easy to transform colourless into yellow crystals. The present paper contains the results of analysis of the two varieties of crystals after further purification. The colourless crystals correspond to the formula $C_2H_2(Cu_2Cl_2)_2KCl$, whilst the yellow ones are represented by $C_2H_2[(Cu_2Cl_2)_2KCl]_2$.

Oxidation of Anethol and Analogous Bodies (Isosafrol, Isosapiol, &c.), containing a Propenylic Lateral Chain.—J. Bougault.—By acting with iodine and yellow mercuric oxide in alcoholic solution on anethol, the latter is oxidised and transformed into an aldehyde, $C_{10}H_{12}O_2$, by fixation of an atom of oxygen. The same operation, made with bodies analogous to anethol, and like it containing a propenylic lateral chain, gives the same results. The author concludes therefore that the oxidation is effected on the propenylic lateral chain. The reaction can be represented by the equation, $C_{10}H_{12}O + HgO + I_2 = HgI_2 + C_{10}H_{12}O_2$. The reaction is quantitative and may serve as the base of a method of estimation of these bodies.

A New Derivative of Benzophenone.—MM. Oechsner de Coninck and Derrien.—The authors investigate the action of several acids, violet and indigo light and ordinary sunlight on benzophenone. By dissolving benzophenone in pure methylic alcohol and exposing it to the sun's rays, beautiful crystals are obtained, melting at about 180° , and having no colour.

Composition of Compounds of Fuchsin with Colouring Matters, Acid by Constitution.—A. Seyewetz.—The author has already shown that compounds formed by the union of acid colouring matters with basic colours, are not only due to the particular nature of the acid groups or the substituted bases in these compounds. He now undertakes a more complete study of these compounds, limiting himself for the moment to those obtained with the well crystalline basic colouring matter fuchsin and acid colouring matters.

MISCELLANEOUS.

A Reaction of Acrolein and some other Aldehydes.—L. Lewin.—For the detection of acrolein the author has found that the following reaction may be made use of:—When piperidine is mixed with a solution of nitroprussiate of soda and acrolein, added either in solution or not, a gentian-blue colouration is produced; this colouration is intense, if the solution is at 0.1 to 1.0 per cent it is a pure blue, at 1/2000 it is perceptible, at 1/30000 the limit is reached, and the solution takes only a greenish colour. The blue colouration becomes violet on the addition of NH_3 , rose-violet on the addition of $NaOH$, greenish-blue with $C_2H_5O.OH$, rusty brown with a mineral acid, and dirty brown on the addition of H_2O_2 . In this reaction the piperidine can be replaced by dimethylamine, but the action becomes less sensitive. Other aldehydes, such as acetaldehyde, paraldehyde, propionaldehyde, and cinnamic aldehyde, give analogous colourations; with acetaldehyde in particular the reaction is very delicate, the limit of sensitiveness exceeding 1 in 12,000. Formic aldehyde, trichloraldehyde, isobutyric aldehyde, benzaldehyde, salicylic aldehyde, phenylacetic aldehyde, cinnanthol, and furfural do not give any colouration with the above reagent.—*Berichte*, vol. xxii., p. 3388.

NOTES AND QUERIES.

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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2123.

ON ELECTRIC TOUCH AND THE MOLECULAR CHANGES PRODUCED IN MATTER BY ELECTRIC WAVES.*

By JAGADIS CHUNDER BOSE, M.A., D.Sc.,
Professor of Physical Science, Presidency College, Calcutta.

(Continued from p. 42).

Mass Action and Molecular Action.

Of the attempts made to explain the action of contact-sensitiveness, Professor's Lodge's theory of coherence has been the most suggestive. The coalescence of water and mercury drops in Lord Rayleigh's experiments, and Professor Lodge's observation of the welding of two metallic spheres by powerful oscillatory discharge in the neighbourhood, apparently lend much support to the theory of electric welding, which explains in a simple manner the diminution of contact-resistance of various metallic filings when subjected to strong electric variation.

On this theory it follows that all imperfect contacts should exhibit a diminution of resistance when subjected to electric radiation. In carrying out a systematic investigation of the contact-sensitiveness of metals, I, however, found that there are substances, of which potassium may be taken as a type, which exhibit an increase of resistance. Potassium is not a solitary instance; I have found a large number of elements exhibiting this action; the number of compounds which exhibits a similar action is also considerable. Other experiments will be presently described which would show that the theory of coherence is inadequate. From the above it would appear that the subject is far more complex than was at first supposed. For various reasons it would be best to distinguish between the two different actions, which may conveniently be described as mass action and molecular action.

Mass Action.—By this is meant the general action, say, between two masses when placed in a very strong electric field. Under the given circumstance, sparking may take place between the bodies, and the two may thus be welded together. From what has been said it will be seen that such action is *non-discriminative*—that is to say, the action will be the same whatever the chemical or physical nature of the substance may be. The best way of showing this action is with drops of liquid, with surface contamination, for any incipient welding will be at once exhibited by the complete coalescence of the drops. The non-discriminative nature of the action is shown in a striking manner in the following experiment. I may mention here that fragments of solid potassium, and in a lesser degree sodium, exhibit an increase of contact-resistance under the action of electric waves. I made a liquid alloy of potassium and sodium, and drops of this alloy were allowed to float on the stratum separating dense Rangoon oil from lighter kerosene, the alloy being of an intermediate density. The drops coalesced when placed in an intense alternating electric field. The next experiment was made with potassium heated under melted (hard) paraffin. By stirring the molten K with a glass rod, the metal was broken up into numerous spherical drops. These also coalesced under similar electric influence. It is, however, to be borne in mind (1) that in the above experiment the substance is in the form of a liquid, and that in this particular condition certain important molecular changes, to be presently described,

cannot very well take place: (2) that the conditions of the experiment are abnormal.

Experiments will be presently described which will show that the observed variation of conductivity produced by radiation is not due to coherence, but to certain molecular changes of an allotropic nature.

Molecular Action.—By this I mean the allotropic modification produced in a substance by the action of electric waves, the allotropic change being due to a difference in the atomic or molecular aggregation. It will be shown that such molecular change does take place by the action of electric waves, and that all the observed effects of variation of resistance of the sensitive substance may be explained on the theory of allotropic transformation due to the above cause. The effect due to molecular changes in a substance is also expected to be modified by the chemical nature of the substance; thus the molecular action due to radiation, giving rise ultimately to the variation of conductivity of the sensitive substance, will be *discriminative*, in contradistinction to the non-discriminative mass action.

Is the effect of radiation due to non-discriminative coherer action or to the discriminative molecular action? That the effect is discriminative, and therefore molecular, appears to be decisively proved by the experiments described below. If further proofs are necessary, they are afforded by the characteristic curves of variation of current with the E.M.F. given by the three types of substances, positive, negative, and neutral; by the continuity of radiation effect on matter; and, lastly, by certain remarkable results I obtained, which show that the effect of ether waves on elementary substances depends on the chemical nature of the substance; in other words, the effect is found to be a periodic function of the atomic weight of the substance. A detailed account of the above will be given in a future paper.

On the Change of Sign of Response in the Receiver, due to a Variation of Intensity of Radiation.

After finding the increase of resistance exhibited by certain substances, I wished to see whether these showed any further difference as compared to substances which exhibit a diminution of resistance. In my determination of the "Index of Refraction of Sulphur for the Electric Ray" (*Proc. Roy. Soc.*, 1895), I used the method of total reflection. I often noticed that just before total reflection, when the intensity of the transmitted beam became comparatively feeble, the receiver indicated an increase instead of the usual diminution of resistance. Professor Lodge mentions in one of his papers that an iron filing coherer exhibits an increase of resistance when acted on by feeble radiation. Thus, if positive substances like iron give a negative reaction, with the diminution of radiation intensity, negative substances may be expected to give a positive reaction with feeble radiation. Very sensitive substances are, however, not so well adapted for an exhibition of this reversed action, possibly because the range of sensibility is comparatively great. But it is not difficult to demonstrate this property in the case of moderately sensitive substances.

The following experiment with a moderately negative substance (arsenic), and a moderately positive substance (osmium), will bring out this interesting peculiarity in a clear manner. The intensity of incident radiation may be varied in two ways—(1) by removing the radiator further and further from the receiver, or (2) by using polarised radiation, whose intensity may be varied by the rotation of an analyser. In the experiment to be described, the first method was adopted.

Experiments with Arsenic Receiver.—A receiver was made of freshly-powdered arsenic. The radiator used emitted radiation of strong intensity. It was at first placed close to the receiver, and there was produced a moderate increase of resistance. It was then removed further and further, and the increase of resistance became less and less. When the distance was increased to 25 cm. the action was reduced to zero. When the distance was

* A Paper read before the Royal Society, February 8, 1900.

increased to 30 c.m. there was a diminution of resistance, showing that 25 c.m. is, in this case, the *critical distance*. The receiver continued to exhibit a diminution until the radiator was removed to a distance of 70 c.m., when the radiation intensity was too feeble to affect the receiver. Now this critical distance may approximately be regarded as a measure of the sensibility of the substance. In this particular case the electric touch has a negative sign. If by any means (some of which will be described later on) the substance becomes more sensitive, i.e., more negative, the critical distance will be increased. On the contrary, if the sensitiveness becomes less (the substance tending towards positive direction) the critical distance will be decreased. The application of this principle is of importance as affording a means of determining the variation of sensitiveness under different conditions.

Experiments with Osmium Receiver.—Substances which are feebly positive give a diminution of resistance when the radiator is close to the receiver, and an increase of resistance when the radiator is beyond the critical distance. Thus the critical distance for an osmium receiver (whose normal action is moderately positive) was found to be about 250 c.m. The radiator at a distance of 300 c.m. produced a deflection of - 3 divisions in a galvanometer placed in the receiver circuit. But at the distance of 200 c.m. the deflection became + 4, and at the reduced distance of 50 c.m. the deflection became + 150 divisions.

In order to avoid confusion, we may choose to call the effect due to strong intensity of radiation as the normal action. The sign of normal action might further be verified, wherever possible, by obtaining a reverse action with feeble radiation intensity.

Molecular Changes produced in Matter by the Action of Electric Waves.

A sensitive receiver made, say, of iron powder, has its conductivity suddenly increased by the action of electric radiation; but the sensitiveness of the receiver is lost after the first response, and it is necessary to tap it to restore the sensitiveness. On the theory of coherence, the loss of sensitiveness is explained by supposing that electric radiation brings the particles nearer, and welds them together, and that the sensitiveness can then only be restored by the mechanical separation of the particles. This supposition, however, fails in the case of substances which exhibit an increase of resistance by the action of radiation. It may, however, be supposed that by some process, little understood, the particles are slightly separated by the action of electric waves, thus producing the observed increase of resistance. On this view, however, the restoration of sensitiveness by tapping remains unexplained. Again, if the increase of resistance is due to a slight separation of particles, suitable small increase of pressure ought to restore the original conductivity, as also the sensitiveness. It is, however, found that a considerable pressure is required to restore the original current, as if the outer layers of the particles were rendered partially non-conducting by radiation, and had to be broken through before the original current could be re-established. It is also found that though the sensitiveness is restored by this expedient of increasing the pressure, yet the restoration is only partial, and that after a repetition of this process the receiver loses its sensitiveness almost completely.

I have attempted to find out an explanation of this obscure "fatigue" effect. This subject will best be treated in connection with the anomalous behaviour of silver, which I find is also in a manner connected with the fatigue effect. Silver, when subjected to radiation, exhibits, as indicated in my last paper, sometimes an increase, and at other times a decrease, of resistance. The difficulty in this case cannot be explained on the supposition of variations of radiation intensity, as the anomaly persists even when the intensity of radiation is maintained uniform by keeping the radiator at a fixed distance.

In order to explain these actions, I assumed the following hypotheses, which, with the necessary deductions, are given below:—

(1). That electric radiation produces molecular change or allotropic modification in a substance.

(2). That, starting from the original molecular condition A, the effect of radiation is to convert it, to a more or less extent, into the allotropic modification B (the latter condition will be designated as the "radiation product"). It follows that this change from one state to the other must be accompanied by a corresponding change in the physical properties of the substance.

(3). As one of the properties of a substance is its electric conductivity, any allotropic changes produced by radiation should be capable of being detected by a variation in the conductivity of the substance.

(4). As a molecular strain is produced during transformation from A to B, at a certain stage there may be a rebound towards the original state A. Thus, after the molecular change from A to B condition has reached a maximum value, the further action of radiation may be to re-convert, to a more or less extent, B to A, this reversal of effect being indicated (see No. 3) by a corresponding electric reversal.

(5). That the ultimate loss of sensitiveness, known as "fatigue," is due to the presence of the radiation product, or strained B variety, along with the A variety, the opposite effects produced by the two varieties neutralising each other.

The justification for the above hypotheses is to be sought for:—

(1). From analogy with other known radiation phenomena.

(2). From experimental proof—

(a). Of the allotropic transformation being attended with changes in the conductivity of the substance.

(b). Of the existence (and, if possible, the production by chemical means) of an allotropic modification analogous to the radiation product or B variety, whose reaction should be opposite to that of the substance in a normal condition (A variety).

(c). Of the assumption that after the maximum transformation of A into B, the further action of radiation is to re-convert, to a more or less extent, the form B into A, such transformations giving rise to electric reversals.

(d). Of the existence of the radiation product in a fatigued specimen.

The above mentioned hypotheses will obtain still stronger support if further deductions from the above theory are borne out by confirmatory experiments.

I will now describe investigations on the lines sketched above.

Allotropic Modification produced by Visible Radiation.

As regards the action of radiation in producing allotropic modification, several such instances are known in the case of visible radiation. In the familiar example of the conversion of yellow phosphorus into the red amorphous variety, the effect is quite apparent. But this is not the case in the transformation of the soluble sulphur into an insoluble variety by the action of light; here the transformation is not apparent, and has to be indirectly inferred from the insolubility of the solarised product in carbon bisulphide. The reason why a far larger number of instances of allotropic transformation produced by light is not known is not because that such effects are not more numerous, but because we are unable to detect such changes. It must be admitted that our knowledge of molecular changes, specially in a solid, and the means of their detection, is at present extremely limited.

Variation of Conductivity produced by Allotropic Changes.

There is one method of detecting these molecular variations to which little attention has hitherto been

given, but which appears to be of great interest, and promises to yield important results in investigations of this class. It is evident that changes in molecular structure must be attended with changes of physical properties, electric conductivity being one of them. Among other instances of allotropic changes attended with changes in electric conductivity may be mentioned the wide difference of conducting power between graphite and diamond. The same great differences of conductivity is seen between the crystalline and amorphous varieties of silicon, and between the "metallic" and white varieties of phosphorus. But it is not at all necessary to take only such extreme cases to show the influence of molecular or atomic aggregation in influencing the conductivity. This effect is brought into painful prominence by the variation produced in spite of all precautions in our standards of resistance.

Experimental Proof of Allotropic Changes being attended with Variation of Conductivity.—I shall now describe a direct experiment by which the change of conductivity produced in a substance by molecular change is exhibited. Red mercuric iodide is converted into the yellow variety by the application of heat, and the substance does not return to its original state till after a considerable lapse of time. The recovery here is very slow. A small quantity of mercuric iodide was now placed in a tube provided with sliding electrodes, and a current was made to pass through the substance by suitable compression. The conductivity of the substance is rather small, and therefore a thin stratum should be taken for experiment. The current is observed by means of a delicate galvanometer. On the application of heat to the tube (which converts the red into the yellow variety), there was at once produced, simultaneously with the molecular transformation, an increase of conductivity. This effect is not due to a rise of temperature, for the increased conductivity was still exhibited on cooling the tube. From this experiment it is seen that the molecular changes can be inferred from changes in the conductivity. In the case described above, the recovery from the B, or second stage, to the first stage, A, is slow; but there may be substances (and there are such substances) where, under the given conditions of temperature and other physical surroundings, the first stage is far more stable than the second; the substance will then pass back quickly from the B condition to the primitive state, on the cessation of the exciting cause, which gave rise to the transient B effect. The substance will in this case be "self-recovering."

(To be continued).

THE ESTIMATION OF LEAD BY ELECTROLYSIS OF THE SULPHATE AND THE CHLORIDES. APPLICATION TO THE ANALYSIS OF LEAD GLASSES AND CHROMATES OF LEAD.

By CH. MARIE.

THE two principal methods for the separation of lead, founded on its precipitation by sulphuretted hydrogen or sulphuric acid in the presence of alcohol, bring it to the form of sulphide or sulphate, which is not adapted to electrolytic estimation on account of its insolubility in dilute nitric acid. As nitric acid easily transforms the sulphide into the sulphate I only considered the case of this latter salt.

To effect the solution of sulphate of lead in nitric acid, I decided after several experimental trials, to use nitrate of ammonia, a reagent which does not introduce any fixed substance, and which can be easily eliminated in the subsequent analytical operations. This solution was made in the following manner:—

The sulphate of lead is placed in the beaker in which it

is to be electrolysed, and attacked with nitric acid to which crystals of nitrate of ammonia are gradually added. To hasten the solution it is heated on a water-bath. When all the sulphate is dissolved the solution is diluted with warm water, and the electrolysis is proceeded with in the ordinary manner, keeping the temperature up to 60–70°. The quantities of the reagents necessary are as follows:—For 0.3 grm. of sulphate it is necessary to have 5 grms. of nitrate of ammonia; as for the nitric acid, its quantity is determined by this condition, viz., that after dilution the liquid ought to contain 10 per cent of free acid.

As the sulphate dissolves more easily in acid which has been slightly diluted than in concentrated acid, it is as well before adding the latter, to pour a little water over the sulphate. In three hours, with an unpolished platinum electrode having a surface of 90 square c.m., and with a current of 0.3 ampère intensity, we can easily deposit 0.4 grm. of binocide of lead.

This method allows of the application of electrolysis to the analysis of lead glass. It is sufficient to attack the finely powdered glass with hydrofluoric acid containing the necessary quantity of sulphuric acid to transform the bases into sulphates. Too great an excess of sulphuric acid will prevent the solution of the sulphate of lead, which should be carried out as described above. After the electrolysis we can proceed immediately to the estimation of the alkaline metals, if the material under examination contains no metal of the iron group, or of the alkaline-earth group.

Estimation of Lead in the Chromates.—The chromates of lead dissolve still more easily than the sulphates in the mixture of nitric acid and nitrate of ammonia. For 0.5 grm. of chromate 2 grms. of nitrate suffice; as for the nitric acid, it is sufficient for the final solution to contain 10 per cent.

The electrolysis is conducted as in the case of the sulphate; the chromic acid, during the operation, is completely brought into the state of sesquioxide of chromium, directly precipitable by ammonia.

By the simplicity of the analytical method and the exactness of the results furnished, this method will be very useful for the analysis of such important commercial products as the silicates and chromates of lead.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No 12.

On Nitracetone.—Ad. Lucas.—Following up a previous publication the author now announces that he has obtained nitracetone in a crystalline form. For this purpose he dilutes the solution of nitrite of silver and iodacetone in ether with a quantity of ether so as to obtain a not too thick liquid; this is kept agitated at 0° until the odour of iodacetone has disappeared, which requires about twenty-four hours. The solution is then filtered and washed with a little ether, then washed a second time with ether in another flask until no residue is left on evaporating the washings to dryness; it is in this second portion that the pure nitracetone is found, while the ether used for the first washing contains the secondary products. On evaporating the second portion of ether, well-formed crystals are obtained, easily soluble in benzene, from which the pure nitracetone separates in the form of beautiful needles. It crystallises in benzene in large plates, is fairly soluble in water, and more easily so in alcohol and ether; it fuses at 49°. The aqueous solution of nitracetone is distinctly acid. The author describes its ammonium salt, $\text{CH}_3\text{CO.CH:NO.ONH}_4$, as well as the phenylhydrazone, $\text{CH}_3\text{C(NH.C}_6\text{H}_5)_2\text{CH}_2\text{NO}_2$. The proof that the substance $\text{C}_3\text{H}_5\text{NO}_3$ obtained by the author is the true nitracetone, $\text{CH}_3\text{CO.CH}_2\text{NO}_2$, or rather the isonitracetone, $\text{CH}_3\text{CO.CH=NO.OH}$, and not its isomer the nitrite of ethylcarbinol, $\text{CH}_3\text{CO.CH}_2\text{O.NO}$, is furnished by the fact that it gives, on reduction, amidacetone $\text{CH}_3\text{CO.CH}_2\text{NH}_2$.—*Berichte*, vol. xxxii., p. 3179.

THE
EIGHTH GROUP OF THE PERIODIC SYSTEM
AND SOME OF ITS PROBLEMS.*

By JAS. LEWIS HOWE.

(Concluded from p. 39):

Of the simple salts of oxy-acids few are known of any metals of this group except the lower series, iron, cobalt, and nickel; a single sulphate of rhodium, one of palladium, and perhaps a double sulphate of platinum, a chromate of iridium, a basic carbonate of palladium, two or three nitrates, a phosphate of rhodium, and a hypophosphite of platinum; such is practically the whole list. The platinum metals have little tendency to form crystalline salts with oxy-acids, and many such salts are unquestionably incapable of existence, but in many cases at least the difficulty is our ignorance of the conditions of formation of such salts. And herein, I may say, is one of the most marked differences between investigation in organic and in inorganic chemistry. In the former the field has been so thoroughly studied that the conditions of reaction are often well known, and the course of a reaction can be foretold with considerable certainty; in inorganic chemistry the work is like exploration in an almost wholly unknown land. We know neither the possibility of existence of conjectured compounds, nor the conditions under which alone such formation or existence is possible. For this reason inorganic research is slower and far more apt to be fruitless. No better example of this can be cited than the fact already referred to, that Professor Joly, as well as myself, exhausted every method which occurred to us for the formation of the tetrachloride of ruthenium, and failed in our efforts by missing just the proper conditions, which happily Professor Antony has hit upon.

But while the platinum metals seem to form few simple salts, few or none show such a decided tendency to form double and complex salts, and this property is, to some extent, shared by the three light metals of the group.

Best known and best developed of these compounds are the cyanides, which are especially familiar to us in the prussiates of iron. In nickel we have the ordinary double cyanide, $K_2Ni(CN)_4$, or $2KCN, Ni(CN)_2$, formed by the solution of nickel cyanide in potassium cyanide. As electrolytically dissociated, the nickel is a positive ion, and the double salt is at once broken up by acids with the precipitation of nickel cyanide. The double cyanide of palladium, $K_2Pd(CN)_4$, is similar but less easily decomposed. The corresponding double cyanide of platinum, $K_2Pt(CN)_4$, is clearly a salt of the complex platinocyanic (or cyanoplatinous) acid, $H_2Pt(CN)_4$, which is formed on treating the salt with a strong acid, can be separated in a pure condition, and is an acid strong enough to expel hydrochloric acid from sal-ammoniac. The platinum atom is here a constituent of the negative ion, $Pt(CN)_4$.

If we proceed from nickel along the horizontal series, we find that while a double cobalt cyanide, $K_2Co(CN)_6$ or $4KCN, Co(CN)_2$, can be formed, it is very unstable, and belongs to the same easily decomposable class as the double nickel cyanide. This cobalt cyanide has, however, a great tendency to oxidise and form potassium cobalticyanide, $K_3Co(CN)_6$, which is stable and a salt of the cobalticyanic acid, which can be obtained in a free state. In passing we note a very interesting point, that under the influence of such reducing agents as potassium cyanide, potassium nitrite, and potassium sulphite, cobalt shows a great tendency to become oxidised from its bivalent condition to the very stable complex compounds in which it is trivalent; under other circumstances, simple compounds in which cobalt is trivalent are formed with great difficulty, and are of decided instability. This

seemingly anomalous property still demands an explanation.

Turning to the iron cyanides we find both types, $K_4Fe(CN)_6$ and $K_3Fe(CN)_6$, ferrocyanide and ferricyanide, well developed and extremely stable. From each, the corresponding acid can be obtained in a free state, and is a strong acid. Of the remaining metals, the double cyanide of rhodium and iridium resemble the cobalticyanides, while of iridium the iridocyanide, $K_4Ir(CN)_6$, is also known and is stable, thus completing the analogy found in the nickel group. Potassium ruthenocyanide, $K_4Ru(CN)_6$, and osmocyane, $K_4Os(CN)_6$, resemble the ferrocyanide, the free acids being easily separable from the salts. Outside of the eighth group, the stable complex cyanides are known only in the case of manganese and chromium.

Regarding the constitution of the double cyanides, you are all familiar with the various suggestions that have been made from time to time, which involve the polymerisation, probably by threes, of the cyanogen group. To this there have been raised two objections: an explanation which is satisfactory for the double cyanides should also be available for the double chlorides, as K_2PtCl_6 , which are also salts of complex acids, and where polymerisation by threes is at least improbable; and second, it is possible to replace a single cyanogen group or chlorine atom without changing essentially the nature of the molecule, as in sodium nitroprusside, $Na_2Fe(CN)_5NO$, and potassium nitrosochlororuthenate, K_2RuCl_2NO . There is a large field for study in these cyanides from the standpoint of the newer physical chemistry.

Closely connected with the chemistry of the cyanides is that of the thiocyanates, but it has been very meagrely worked out for the eighth group. In the case of platinum both potassium platino- and platithiocyanates, $K_2Pt(SCN)_4$ and $K_2Pt(SCN)_6$ are known, and are salts of the platino- and platithiocyanic acids. These are complex acids and may be separated out, but in the free state are very unstable. The double ferric thiocyanates may be formed, but there is no corresponding complex acid, that is, they are ordinary double salts. The ferrous, cobaltous, and nickel thiocyanates are known, but form no double salts. It is extremely probable that the other metals of this group would show a full series of thiocyanates.

Another interesting class of complex salts is that of the double nitrites, first studied in the case of platinum by Nilson, but for the other platinum metals by Wolcott Gibbs, who bases upon these his method of separating the metals. More recently these nitrites have been investigated by Joly, Vèzes, and Leidé. The most familiar double nitrite is the potassium cobaltinitrite, which has long served for the separation of cobalt from nickel, and which is also used as a pigment under the name of aureolin or cobalt-yellow. These nitrites resemble, to a considerable degree, the double cyanides, and in the case of iridium the free complex iridonitrous acid has been obtained; in the case of iron, cobalt, and nickel, we have also representatives of a large class of very stable triple nitrites, first noted by Künzel and Lang, and studied by Erdmann (*J. Prakt. Chem.*, 1866, xcvi., 385). More recently these have been investigated by Przibilla (*Ztschr., Anorg. Chem.*, 1897, xv., 419), who, after great difficulty, succeeded in preparing the triple iron potassium nitrites with lead, barium, strontium, and calcium: this is the first nitrite of iron to be prepared, and leaves osmium as the only metal of the eighth group of which no nitrite is known.

In the case of all the platinum metals double sulphites are known, which are salts of complex metallo-sulphurous acids. In these metals the presence of the sulphurous acid radical cannot be detected by ordinary reagents. In the case of cobalt a full series of cobaltisulphites is known, which are stable salts, while the cobaltosulphites are very unstable. Little is known of iron and nickel sulphites, and there is much room for further investigation in the case of the sulphites of the other elements of this group.

* An Address delivered before the American Association for the Advancement of Science, New York Meeting (1900), Section C.

There is at the same time reason to believe that a study of the thiosulphites and possibly the dithionates of this group would not be without interest.

Another acid which is capable of forming complex salts is oxalic. The platoxalates are the only ones which have been carefully studied, though some work has been done upon the rhodoxalates. Several iron oxalates and double oxalates are known, but aside from this the field is unworked but promising. In this connection it may be added that while oxalic acid is the only organic acid which has been investigated to any considerable extent in complex salts, it by no means follows that it is the only acid which is capable of entering into such combinations. Some of my students have made preliminary tests with a large series of acids and found that several among them enter combination with chromium with the formation of complex salts, and it is quite possible that similar compounds may be formed with the eighth-group metals. Mention should also be made that Gibbs has introduced platinum into his complex salts, forming platinimolybdates and platinitungstates.

Since complex salts of hydrocyanic, nitrous, sulphurous, oxalic, and other analogous acids, are best developed generally with the metals of this group, it is in the study of these and other compounds of this group that we may hope to gain an insight into the constitution of these interesting compounds of which so little is known, and further extend our knowledge regarding valence, for it is just at this point that the generally accepted theory of valence begins to break down.

Before alluding to the ammonia bases, which are so well developed in this group, and would naturally follow these complex salts we have just considered, a brief digression may be made to refer to three classes of anomalous compounds, which should not be passed without reference. The first of these are the nitroso compounds. It is only recently that, largely through the efforts of Joly, the nature of the so-called nitro prussides was discovered,—double cyanides in which one cyanogen group is replaced by one nitroso group, NO. Joly then found that the old osmiamic acid of Fritzsche and Struve is also to be considered as a nitroso compound, and that the supposed tetrachloride of ruthenium is, in reality, a nitroso-chloride. But while there appear to be no representatives of the nitroso compounds in the cobalt or the nickel groups, several other compounds of iron are known which contain this group, as the potassium iron tetra- and heptanitroso-sulphonates, $K_2Fe_2(NO)_4S_2$, and $KFe_2(NO)_7S_3$, and the iron nitroso-thiocarbonate and thio-antimonate of Löw. There seems also to be a nitroso-cyanide of ruthenium, corresponding to the nitroprussides, but it has not been isolated. In none of these cases has the interesting question been brought out as to whether the nitroso group remains attached to the metal when in solution, or whether it is electrolytically dissociated and acts the part of an acid radical.

In some respects yet more remarkable are the compounds formed with carbon monoxide and with phosphorus trichloride. The best known compound of this class is the nickel carbonyl, $Ni(CO)_4$, of Ludwig Mond. The nature of this volatile liquid is yet unknown, but it is by no means unique. Immediately after its discovery it was found that iron formed similar compounds, $Fe(CO)_5$ and $Fe_2(CO)_7$. That a volatile compound of iron exists had been very apparent on the lime of the Drummond light, when water-gas, compressed in iron cylinders, was used instead of hydrogen, and also in the clogging of gas burner tips with an oxide of iron, especially when a carburetted water-gas is used as an illuminant. The volatile iron carbonyl seems to be formed at ordinary temperatures by the passage of carbon monoxide through iron pipes. But it is not alone with metals that carbon monoxide combines to form volatile compounds. As early as 1868 Schützenberger (*Comptes Rendus*, 1868, lvi., 666, 747) discovered that platinous chloride, $PtCl_2$, would combine directly with carbon monoxide, with the formation of

three distinct compounds, containing respectively one, two, and three molecules of CO to one of $PtCl_2$. A compound is also known in which one CO group replaces one cyanogen group in potassium ferricyanide, that is $K_3Fe(CN)_5CO$. This reminds us naturally of the nitroso-ferricyanide, the so-called nitroprusside. Again in 1870 Cahours and Gal (*C. R.*, 1870, lxx., 897, 1380; 1870, lxxi., 208) discovered a series of compounds containing platinous chloride united with phosphorus trichloride, and also with some of the organic phosphines. These compounds are not of the nature of double chlorides, for they can be hydrolysed with the formation of chloroplato-phosphorous acid. An analogous class of compounds of iridium has been made by Geisenheimer (*C. R.*, 1890, cx., 1004), which are also capable of hydrolysis, giving chlor-iridophosphorous acid. Geisenheimer has formed similar compounds containing bromine (*C. R.*, 1890, cx., 40) in the place of chlorine, and also others containing arsenic (*C. R.*, 1890, cx., 1336) in the place of phosphorus. How far compounds of this nature can be extended is only conjectural, but there is evidence of the existence of something of the kind with iron.

Of binary compounds with the less negative elements, such as the phosphides and carbides of iron, little is known. Like iron, nickel and also platinum and iridium form phosphides. Iridium phosphide possesses an economic importance in that it enables the metal to be fused in a furnace. Up to the discovery of this process in 1882 by Dr. Wm. L. Dudley, the native grains of iridosmium were alone available for tipping gold pens, stylographs, and the like. It was, however, found that when iridium was heated to a high temperature in a crucible, on introducing a piece of white phosphorus, the whole mass immediately melted, and could be cast into plates, afterward to be worked up into desired form. This reminds one of the early method of working platinum by alloying it with arsenic and then roasting the arsenic off in a muffle.

There remains a single class of compounds to be noticed, the ammonia bases, whose greatest development is found in this group. The first member of this class was the compound now known from its discoverer as the green salt of Magnus, which was first made in 1828 (*Ann. der Phys.*, Pogg., 1828, xiv., 239). Then came the work of Gros, of Reiset, and of Peyrone. Among the many chemists who have cultivated this field are Clève, Jörgensen, who has given us most of our knowledge of the rhodium bases, Gibbs, Palmaer, who has developed the iridium bases, and Joly, who has revised the bases of ruthenium; while the theory of these bases has been discussed especially by Claus, Blomstrand, Jörgensen, and Werner. In connection with these bases, appear what must with our present knowledge be considered anomalies. The greatest development of these bases is found with platinum, where nearly or quite a dozen distinct classes of bases are known, and where we find several groups of isomers, which Werner seeks to explain as stereoisomers, while Jörgensen strenuously combats the view. In type, the palladium bases resemble those of platinum, but as far as yet studied are much less well developed. Nickel, on the other hand, forms no true bases, though many ammonia compounds. The cobalt, rhodium, and iridium bases are all formed on the same general types, but by far the greatest development is found in cobalt, which almost rivals platinum in the number of classes; but few of these are developed with iridium, and fewer still with rhodium. In the iron group no bases are formed by iron, and only two or three ammonia compounds; ruthenium and osmium form fewer bases, as far as yet investigated, than any of the other platinum metals. It is interesting to note, however, that one of these ruthenium bases, discovered by Joly, which possesses intense tinctorial power, resembles very strongly an organic dye, both on fabrics and as a stain in microscopy. The constitution of the ammonia bases is to-day, as it has been for half a century, one of the greatest problems of inorganic chemistry, and it is apparently no

nearer solution. In accordance with the valence theory, it becomes necessary with Jørgensen to assume the existence of chains of at least four NH_2 groups in a molecule, stable enough to be unaffected by aqua regia, and also that these ammonia groups are replaceable by water molecules. We must also assume that while in ordinary salts, as, for example chlorides, the chlorine atoms, which are directly united with the metal, are dissociated in aqueous solution, in these bases the chlorine which is directly united with the metal is not dissociated, but that which is united with the metal through the medium of one to four ammonia groups is dissociated. Led by a consideration of these seeming inconsistencies, Werner has proposed his theory of co-ordinated groups within the molecules,—a theory which seems to possess at least elements of truth, even if not expressing the whole truth. It is possible, too, that Werner's theory may explain some of the difficulties of the theory of electrolytic dissociation, and harmonise it with the hydrate theory of solution.

The constitution is, however, not the only problem of these bases. To my mind their connection, or rather lack of connection, with the periodic system, is one of the most inexplicable facts in chemistry. It makes it apparent that while the periodic law expresses a truth, without doubt the greatest generalisation of modern chemistry, yet even this is in its present statement not the whole truth. We find a marvellously full development of these bases in connection with cobalt, platinum, and chromium. Manganese and iron which lie between chromium and cobalt form no bases. The higher members of the chromium group—that is, molybdenum, tungsten, and uranium—form no bases, while the higher members of the iron series—that is, ruthenium and osmium—do. Of nickel, which stands next to cobalt and resembles it so closely, no bases are known, and yet it is the lowest member of the series which contains platinum. It is true that bivalent cobalt forms perhaps, like nickel, no bases, but as trivalent cobalt forms so many bases, trivalent iron would seem likely to form many, instead of none. If indeed manganese and iron are capable of forming these bases, it seems strange that no one has yet happened upon the proper conditions. It is the consideration of a subject like that of these inorganic bases which forces upon us a realisation of how much there is after all which we do not know about chemistry.

We turn now to a short consideration of the eighth group from a theoretical standpoint. Following Dr. Venable (see Periodic Table, p. 16) we may assume that each of the first seven groups consists of a group element, as in group one, lithium; a type element, as sodium; and two series, one of more positive elements, as potassium, rubidium, and caesium, and the other more negative, as copper, silver, and gold. Further, the more positive the type metal, the more closely will the metals of the positive series resemble it; the more negative the type metal, the more closely will the negative series resemble it. Thus in the first group, the positive series potassium, rubidium, and caesium closely resembles the type element sodium; in the seventh group the negative series, bromine and iodine, resembles the type element chlorine. Now the eighth group differs materially from the other seven in that it contains three series, with no group or type element. These three series are transitional from the least positive among the seven positive series, manganese, to the least negative among the negative series, copper, silver, and gold. The properties of the metals of group eight show this transition as from a chemical standpoint; iron, cobalt, and nickel form a direct gradation between manganese and copper. Now comes a further question as to possible transition elements between the most negative series, fluorine, chlorine, bromine, iodine, and the most positive series, sodium, potassium, rubidium, and caesium. From a theoretical standpoint such transition elements should be neither positive nor negative, and should have a valence of zero. A few years ago the realisation of such a conclusion would have seemed impossible, yet,

since the discovery of argon and its congeners, it seems almost probable that these places have been filled in accordance with theory. If we take the most generally accepted atomic weights, we find helium preceding lithium, neon following fluorine and preceding sodium, and argon really between chlorine and potassium, but with an atomic weight apparently slightly greater than that of potassium which follows it, resembling in this respect cobalt and nickel of this same group, and also tellurium and iodine. There would, in addition, be expected from the analogies of group eight, one, two, or three transitional elements between bromine and rubidium, of atomic weight 80 to 85, and Ramsay has suggested that krypton may belong in this place—so also an element or elements of similar character might be expected between iodine and caesium, with atomic weight of about 130. The recently published work of Ladenburg and Kruegel on krypton give it an atomic weight of about 59. This would, as Professor Ladenburg suggests, make it immediately precede copper; but unless we change very materially our ideas of the periodic law, it is difficult to conceive of an element with the properties of krypton lying between nickel and copper. If these inert gases belong in the eighth group it may seem strange that iron and the other familiar metals which belong here should be so unlike such a type element as argon or neon; it must, however, be borne in mind that this is only an expected exaggeration of the departures found in the first and seventh groups, where copper departs from its type element sodium, and manganese from its type element chlorine. As to whether three elements are to be expected of atomic weight 130 between the light and the heavy platinum metals, we have little data upon which to theorise. As a matter of fact there is very little definite knowledge of the elements between cerium and tantalum. The inter-Jovian planet proved to be an indefinitely large number of asteroids; Sir William Crookes's study of the rare earths leads him to the conception of a group of asteroidal meta-elements in this vacant space in the periodic table. We must await further knowledge before these problems can be satisfactorily solved.

In conclusion, one word as to a very practical problem connected with this group. It is but a few years past a century since the use of platinum was introduced into the chemical laboratory. For a few decades the supply exceeded the demand, but the applications of platinum have steadily increased, and never so rapidly as in the last two decades. For many purposes no substitute for platinum has been found. At the same time the supply of platinum is not keeping pace with the demand, and as a result the price of platinum has very materially advanced. While platinum is very widely distributed, there are few places where it occurs in workable quantities. It is possible, however, that it has been often overlooked, as in placer mining for gold, and efforts have been made to attract miners' attention to more careful search for platinum deposits. At the present outlook it will, within a few years, be imperatively necessary either to materially increase the platinum supply of the world or to replace it for many purposes by some other substance. How this problem will be solved cannot now be foreseen.

On Lilienfeld's Synthetic Peptone.—M. Klimmer.—Lilienfeld has prepared a substance which he considers to be peptone, by the condensation of phenol and glycol by means of oxychloride of phosphorus, or by the condensation of asparagin and para-amidobenzoic acid in the presence of glacial phosphoric acid or metaphosphoric acid, &c. In taking up this subject M. Klimmer has shown that Lilienfeld's conclusions are incorrect; in fact, the substance obtained differs from peptone in at least two characteristics, it does not show the biuret reaction, and it decomposes with the greatest ease into its constituents.—*Pflüger's Archiv*, vol. lxxvii., p. 210.

THE
ESTIMATION OF PHOSPHORUS IN STEEL, &c.

By FRED IBBOTSON and HARRY BREARLEY.

A BEWILDERING number of papers have been written on the estimation of phosphorus in steel. The following process is described only because it is believed to be quite new in respect to the means of estimating the phosphorus.

In iron and steel works phosphorus is almost exclusively separated as ammonium-phosphomolybdate. Mainly for the sake of dispatch, it is either weighed in that form, or the MoO_3 estimated volumetrically after reducing with zinc, or by saturating with standard alkali.

The percentage of phosphorus in the yellow precipitate, says Blair, is given by various authorities from 1.27 to 1.75. Both Blair ("Chem. Anal. of Iron," 2nd edition, 1891) and Clowes and Coleman ("Quant. Anal.") give the formula $(\text{NH}_4)_3\text{PO}_4 \cdot 11\text{MoO}_3$ to the precipitate dried at 120°C .; Arnold ("Steel Works Analysis") gives the same formula plus nine molecules of water when dried at 100°C ., and each compound is said to contain 1.63 per cent P. Really the formula $(\text{NH}_4)_3\text{PO}_4 \cdot 11\text{MoO}_3$ corresponds to 1.79 per cent P.

Hundeshagen (CHEM. NEWS, lx, 168)—who has made the most complete examination of the formation of the yellow precipitate we have seen—and many others write the formula $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3$ when the precipitate is dried at 120 – 150°C . This formula has been adopted in the calculations of the following process.

It has been previously shown (CHEM. NEWS, lxxix, 4) that if we add to a solution containing Mo and P enough HCl to prevent the formation of a precipitate, on adding at least enough lead acetate to combine with the molybdic and phosphoric acids as PbMoO_4 and PbHPO_4 , and then add this heated mixture to another containing ammonium chloride and at least enough ammonium acetate to decompose the free HCl, we get PbMoO_4 precipitated and PbHPO_4 in solution. The results are exact when much greater proportions of P than P : 12MoO_3 are present. The new process depends on these facts.

Various amounts of dried yellow precipitates prepared from soda phosphate were treated in the way indicated. The results were:—

Grm. P calculated from—	
$(\text{NH}_4)_3\text{PO}_4 : 12\text{MoO}_3$	PbMoO_4
0.00041	0.00039
0.00082	0.00082
0.00164	0.00163
0.00246	0.00247
0.00410	0.00393

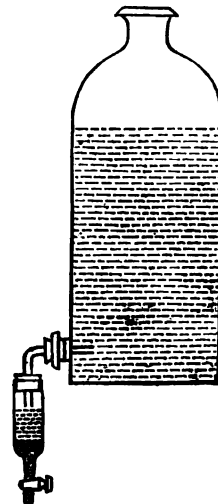
An outline of the process applied to steels is as follows:—Dissolve 2 grms. of the sample in 45 c.c. 1.20 HNO_3 ; add permanganate until a pink colour or an MnO_2 precipitate persists after a few minutes' boiling; clear with FeSO_4 , add about 4 c.c. strong ammonia, and then to the clear hot solution 30 c.c. of the molybdate reagent. Shake the stoppered flask, allow to stand a few minutes at 70 – 80°C .; pass through small pulp filter, wash, dissolve off yellow precipitate with a few drops of ammonia, clearing the sides of the flask with the same solution as it drops from the end of the funnel; pass solution again through the filter into a small beaker (200 c.c.), and wash. Add 10–12 c.c. HCl, 10 c.c. lead acetate (40 grms. per litre), and heat. In the washed-out flask heat a mixture of ammonium chloride (= 10–12 grms. $(\text{NH}_4)\text{Cl}$) and 50 c.c. strong ammonium acetate; mix the

two solutions in the flask with shaking, filter, and weigh as PbMoO_4 . The weight multiplied by 0.007 gives the weight of P in the steel. The operation is very conveniently performed with batches of four, which are completed in an hour and a half. A single estimation takes about forty minutes.

Some noteworthy features of the process are:—The PbMoO_4 is quite insoluble in the solutions used; being very granular it filters readily; it can be ignited speedily without loss of weight, and it weighs more than one hundred and forty times heavier than the phosphorus to be estimated.

Although it simplifies the process to use constant amounts of reagents, still some variation is possible without greatly sacrificing accuracy. For instance, a series of two separate steels were in each case dissolved in 45 c.c. HNO_3 , but varying amounts of strong ammonia were added, and the process concluded as usual. The results were—

$(\text{NH}_4)\text{HO}$	Per cent P.	
	I.	II.
0	0.088	0.032
2	0.090	0.032
4	0.090	0.034
6	0.089	0.034
8	0.091	—



The process has been satisfactorily applied to the estimation of phosphorus in pig-irons, nickel-steel and alloys, chrome-steels, tungsten-steels, spiegels, and ferro-manganese. The graphite of pig-iron should be filtered off before adding the permanganate. Chrome-steels require more permanganate, because the Cr is oxidised to CrO_3 and de-oxidised by the FeSO_4 . Spiegels, FeMn, and tungsten steels also need more permanganate. Very little WO_3 is precipitated during the oxidation; it is well, however, to pass through a filter before adding the molybdate. A little WO_3 is sometimes precipitated with the phospho-molybdate, and gets weighed finally as PbWO_4 , but in extreme cases it influences the result only to about 0.002 per cent P. It is detected by dissolving the lead molybdate in a few c.c. HCl, evaporating, and taking up with dilute HCl. The WO_3 remains undissolved.

As it is important that no MoO_3 , deposited from the reagent or dried on the neck of the bottle, should pass into the solution of the sample, the ammonium molybdate is filtered as required through asbestos felt arranged in the throat of a stopcocked cylinder funnel, as shown in the sketch.

The Technical Department,
University College, Sheffield.

* In 1895 Blair and Whitfield (Journ. Am. Chem. Soc., xvii., 747), after analysing ten different samples of the yellow precipitate, fixed 1.794 as the ratio—

$$\frac{\text{P}}{100\text{MoO}_3}$$

which agrees with the ratio calculated from Hundeshagen's formula.

LABORATORY APPARATUS.

A STEAM-PIPE CONDENSER AND A GLASS TUBE OVEN.

By F. W. STREATFEILD, F.I.C.,
and
F. SOUTHERDEN, F.I.C.

FIG. 1 shows the first piece of apparatus as set up for work in the laboratory. It is intended for the distillation of substances which tend to solidify in the condenser. To obviate this, a glass tube runs the whole length inside, and is connected to the steam generator by a rubber tube and pinchcock. When a block threatens, the cock is opened and the heat from the steam rapidly removes any obstruction.

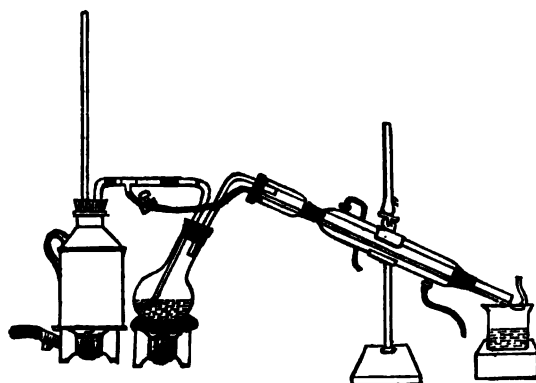


FIG. 1.

This method is quicker and more under control than the ordinary one of letting the water out of the condenser. It is necessary that the condenser should possess a wide mouth, so that the two tubes may be readily inserted.

Before proceeding to make a determination of the melting-point of an organic compound, it is most essential that any adhering solvent which may have been used in its purification be removed. This may be effected by placing the substance in the water- or air-oven. If the drying is conducted in a current of hot air, the operation of complete drying is more quickly carried out. The little contrivance seen in Fig. 2 is for this purpose.

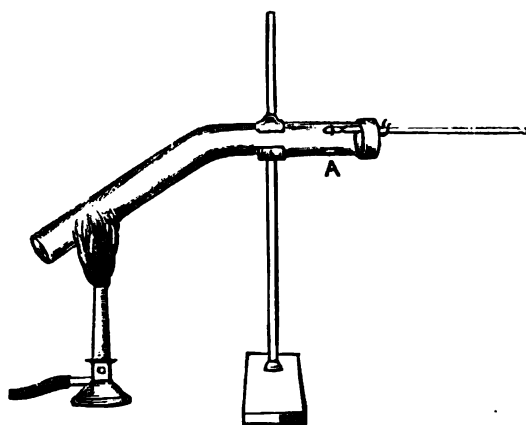


FIG. 2.

A piece of combustion-tube about 35 c.m. in length and 18 m.m. internal diameter is bent, as shown, at an angle of 140°. It is clamped with the shorter arm nearly horizontal, and in this is placed the substance (at A) on a small piece of drying paper. A wire is twisted round the

short arm and bent over the end to form a loop, through which passes a thermometer, the bulb being over the sample. The thermometer maintains its position by its own weight, but to prevent actual contact with the glass tube a piece of fibrous asbestos is wrapped round the bulb. If this is done with wet asbestos no difficulty will be encountered in making it adhere. The substance is dried by a current of hot air produced by the Bunsen burner. This may be placed at any height and at any point along the longer arm, the temperature varying accordingly.

The principal features of the apparatus are that the range of temperature is practically unlimited, and that, without any precautions, the thermometer readings may be kept constant within a few degrees.

With a tube of the above dimensions, the thermometer will always be 10° or 15° higher than the substance, so that the temperature may be safely maintained at its melting-point without fear of melting actually occurring.

and Guilds Technical College,
Finsbury.

ON THE
COMPOSITION OF N. S. WALES LABRADORITE
AND TOPAZES, WITH A COMPARISON OF
METHODS FOR THE ESTIMATION OF
FLUORINE.*

By G. HARKER, B.Sc.

Analysis of Mineral Labradorite from N. S. Wales.

THE specimen described in the following note was collected by Mr. D. A. Porter, of Tamworth, on Sandilands Mountain, some 57 miles from Tenterfield, who forwarded it to Professor Liversidge for examination. Mr. Porter states that it occurs there in a basalt, and also between Hillgrove and Grafton.

The specimens were in broken fragments about half an inch in diameter. Some were colourless, others brown to greyish. One perfect cleavage was exhibited, probably basal, and a second not quite so good. The fracture was uneven, inclining to conchoidal; the lustre vitreous. The hardness was 6 and the sp. gr. 2.70. Before the blowpipe it fused to a colourless glass, its fusibility being about 3. All these properties agree in every detail with those given in Dana for typical labradorite, as do all the optical properties which were determined. The refractive index was low and the double refraction weak. In convergent polarised light a cleavage flake gave a figure with one brush nearly straight, and was therefore the section of a bi-axial mineral cut at right angles to the optic axis, the optic axes being nearly at right angles. The dispersion could not be obtained. The mineral was optically positive and showed multiple twinning.

Analysis.

	1.	2.	3.	4.	5.
	New South Wales.	Veltin.	Thann- bergthal.	Ramoni- Brod.	
Silica	55.05	54.81	55.15	53.61	54.55
Alumina	30.15	29.70	29.15	29.68	28.68
Ferric oxide ..	10.32	0.42	—	—	1.03
Lime	5.11	9.61	9.90	10.96	11.23
Soda	None	Undeter.	5.23	4.36	4.62
K ₂ O	None	0.20	0.80	1.15	0.48
MgO	None	0.28	—	—	—
H ₂ O on ignition	Undeter.	0.13	0.67	0.65	—

100.63 Incompl. 100.90 100.41 100.53

Nos. 1 and 2 are analyses of two different samples of the mineral. In the first analysis the ferric oxide was

* Read before the Royal Society of N. S. Wales, December 6, 1899.

not determined separately; 30.15 is the percentage of mixed oxides of iron and alumina; the analysis was done on the dried ignited sample. Nos. 3, 4, and 5 are three analyses of labradorite given in Dana, and are put in for sake of comparison. The silica in the labradorite analyses given in Dana varies from 54.45 to 56.18, the alumina from 27.33 to 29.85, the lime from 9.90 to 12.01, and the soda from 3.90 to 5.23. From these it will be seen that the chemical composition of the mineral agrees well with that of labradorite, and taken in conjunction with its physical properties shows that it is that mineral.

Topaz.

The following investigation was also made in the Chemical Laboratory of the University of Sydney at the suggestion and under the direction of Professor Liversidge, with the object of determining the composition of some N. S. Wales specimens, and of comparing certain of the published methods for the analysis of fluorides. The most difficult constituent to analyse in topaz is fluorine, for the estimation of which a number of methods have been proposed. To find the most satisfactory was the first object in view.

Before proceeding farther a *resumé* of previous methods for the determination of fluorine might be of use:—

(a) *Fluorides not decomposable by Sulphuric Acid.*—Berzelius was the first to accomplish the analysis of fluorides undecomposable by sulphuric acid. He fused the fluorides with alkaline carbonates. To separate the silica from silicates combined with fluorine he employed a solution of zinc carbonate in ammonia; zinc silicate is formed, and the solution is freed from the rest of the zinc oxide by evaporation.

H. Rose in 1849 separated the fluorine as calcium fluoride, a more convenient form than the sodium fluoride employed by Berzelius. He precipitated the calcium fluoride together with calcium carbonate to help in its filtration. He pointed out that some fluorides which were not associated with silica were not completely decomposed by fusion with alkaline carbonates. A combination of the methods of Berzelius and Rose is still the best method known for the estimation of fluorine. Wöhler in analysing topaz fused with sodium carbonate alone.

(b) *Fluorides decomposable by Sulphuric Acid.*—For the analysis of these a large number of methods have been proposed; Wöhler added silica and sulphuric acid, weighed, and determined the fluorine by loss due to escape of silicon tetra-fluoride.

Liversidge (CHEMICAL NEWS, 1871) decomposed with silica and sulphuric acid, and collected the silicon tetra-fluoride in ammonia solution. By partial evaporation the gelatinous silica was dissolved. He then added potassium chloride, and weighed the fluorine direct as potassium silicon fluoride.

1872, Fresenius collected the silicon tetra fluoride in tubes containing moistened pumice.

Penfield (*Journ. Chem. Soc.*, 1879) passed the silicon tetra-fluoride into a solution of potassium chloride and alcohol, and titrated the hydrochloric acid set free by the reaction.

Carnot (*Comptes Rendus*, 1893) collected the silicon tetra fluoride in a 10 per. cent solution of potassium fluoride, and weighed as potassium silicon fluoride.

Three of the above methods were tried for the estimation of fluorine in topaz:—

First Method, Fusion with Alkaline Carbonates.—The topaz first analysed was from Mudgee, N.S.W. It was in the form of transparent rolled pebbles of from one-quarter to one-eighth of an inch in diameter. The method of analysis first used was that of Wöhler, as given in his "Mineral Analysis," English edition of 1871.

"The substance, when fused with four times its weight of anhydrous carbonate of soda, is decomposed with formation of fluoride of sodium, which is extracted with water. Before filtering off the residual silicate of alumina, the

solution should be digested with carbonate of ammonia to precipitate any small quantities of alumina and silica which may have been dissolved." The residue is evaporated down with hydrochloric acid, and the silica and alumina extracted. The fluorine in solution is removed as calcium fluoride by adding calcium chloride.

This method, when tried on the topaz crystals from Mudgee, gave results for fluorine which were low and irregular, viz., from 5 to 11 per cent. On treating some Brazilian topaz later in the same way, similar unsatisfactory results were obtained, viz., 4.7 to 12.9 per cent.

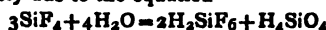
Second or Tetra-fluoride Method.—That of Liversidge as given in Crookes's "Select Methods," p. 580, 1884 edition.

In this method the substance is decomposed with sulphuric acid and silica, and the silicon tetra-fluoride passed into solution of ammonia. The mixture is heated in a platinum retort, first over the water-bath and finally at 160°C., the last traces of gas being removed by a current of air. The ammonia solution is evaporated until the gelatinous silica passes into solution, and is then precipitated as potassium silicon fluoride by potassium chloride and alcohol.

So far this method had only been used for the determination of fluorine in coprolites and apatite. In order to make the method applicable to topaz, which is not decomposable by sulphuric acid, the topaz was first fused with sodium potassium carbonate, silica was then added, and the mixture decomposed with sulphuric acid.

Preliminary trials were made with calcium fluoride, apatite, and cryolite, the substance in each case being fused with 2 or 3 grms. of sodium potassium carbonate and then decomposed. The silicon tetra-fluoride was collected in water after the first few experiments, as it was found that on evaporation with ammonia some of the hydrogen silicon fluoride was converted into silica. The method gave uniform results, which, however, were rather low in all cases.

The cause of the lowness in the results is not apparent. It is probably due to the equation—



not being a quantitative one. Potassium silicon fluoride was found to be slightly soluble in a 50 per cent solution of alcohol. After filtering off the gelatinous silica, the solution in which the potassium silicon fluoride was precipitated was always made of the same volume, and hence the error due to solubility could be allowed for. Potassium chloride was used prepared from different sources, but caused no difference in the results.

The discrepancy was not due to incomplete absorption of silicon tetra-fluoride in the water. A second absorption-tube gave no trace of a precipitate. To completely remove the gas from the decomposition flask, dry air was passed through for four to five hours.

The amounts of calcium fluoride used were varied to see if the error was constant or proportional. The percentage of fluorine obtained was increased as more calcium fluoride was taken, but not proportionally, and tended to reach a maximum. For amounts approximately equal the results were constant, and hence the error can be corrected from the weight of potassium silicon fluoride obtained.

(To be continued).

Synthesis of $\alpha\alpha$ -Dimethyl- γ -cyanotricarballylic Ether and $\alpha\alpha$ -Dimethyltricarballic Acid.—A. Haller and G. Blanc.— $\alpha\alpha$ -Dimethyltricarballic acid, $\text{C}_8\text{H}_{12}\text{O}_6$, is one of the numerous products of degradation which are obtained as a result of oxidation of certain terpenic derivatives, in particular those which are allied to pinene and camphene. The synthesis of this substance therefore presents a certain importance from the point of view of the determination of constitutional formulæ in this series. —*Comptes Rendus*, cxxxi., No. 1.

CORRESPONDENCE.

THERMAL CENTRES OF STABILITY.

To the Editor of the Chemical News.

SIR,—It appears that a confusion has arisen in regard to the sense in which I have used the words "heat of formation." It is certainly not a good term, as it does not express clearly the idea of elevation or degradation of the potential energy which usually accompanies the change of one system into another.

The following, I think, should make my meaning clear:—Imagine a chemical system A. Let the *absolute amount* of energy contained in it at T° and at constant volume be E_A . Now, suppose by some means the system A is converted *isothermally* into another system B; the volume remaining constant. Let the quantity of energy which must be either supplied to, or abstracted from, the system A in order to convert it into B be denoted by $\pm q$. Then—

$$E_A \pm q = E_B,$$

or—

$$\pm q = E_B - E_A.$$

Here q denotes what I have called the "heat of formation," it is simply the energy that must be supplied to one system to "form" the other system from it; q is negative or positive, according as $E_B \leq E_A$. The term "negative heat of formation," if used merely to express the degradation of potential energy brought about by the change, becomes intelligible.

As Mr. Hill points out, if used in an absolute sense, it becomes incomprehensible. Mr. Hill's suggestion that for "negative heat of formation" the term "minimum heat of formation" be substituted, would bring out the nature of the physical changes involved very clearly. In the mathematical treatment of the subject, however, we only deal with the elevation or degradation of energy. It would perhaps be better to avoid the term "heat of formation" altogether, and use "heat of reaction" or "heat toning."—I am, &c.,

GEOFFREY MARTIN.

13, Hampton Road, Bristol.

THE ACTION OF IRON ON WATER.

To the Editor of the Chemical News.

SIR,—It seems to me astonishing that our modern standard text-books on chemistry do not mention the fact of the readiness with which ordinary iron decomposes water at ordinary temperatures with an evolution of hydrogen gas. In our elementary work here, when studying the effect of water on metals, it is always a source of much interest to observe from day to day the *continued* effect of water on the commoner metals. In the case of iron, when a test-tube of filings is covered with water, "red-rust" first occurs on the surface, and then beneath this rust appears the greenish-black oxide in a flocculent form (this is referred to later on), and finally, bubbles of hydrogen. By suitably arranging an apparatus, a volume of gas may be collected in two weeks, using iron filings, or in a few hours if *ferrum reductum*, B.P., be used, sufficient to demonstrate its properties. The same is true if zinc dust be used. Distilled water recently boiled and cooled, I find shows an evolution of hydrogen sooner than common water, and no trace of red rust is observed if air be excluded.

The experiment, lasting as it does over a fairly long interval of time, centres an interest which is so often lost in those of a shorter duration, and it seems to me most instructive, especially when afterwards taken in conjunction with the action of sodium on water.

Although numbers of papers have appeared on the

above subject, both in English and German, yet our modern English text-books pass it over, and also the fact that the oxide first formed contains ferrous iron. This was shown to be the case by Liversedge (*CHEM. NEWS*, lvi., p. 230).

Many experiments have been carried out here quite recently, using different grades of iron and various waters, but I find most of the results have already appeared in German papers.

Many teachers I have spoken to on the subject seemed to be quite unaware of the fact that hydrogen gas was evolved at ordinary temperatures, most of the books either stating there is no action, or that the gas is slowly evolved at 100° .—I am, &c.,

WM. FRENCH.

Bury Grammar School.

July 21, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxi., No. 1, July 2, 1900.

The Combustible Gases of the Air. Air in Woods, Air on High Mountains.—Armand Gautier.—The author continues his researches on the proportion of hydrogen and the combustible hydrocarbons in the air. His experiments were conducted in a wood containing oak, pine, and birch trees at Lainville. He found that the air still contains both hydrocarbons and hydrogen. In order to answer the question as to whether these are necessary constituents of the atmosphere or whether they disappear in very pure air, he continued his researches on Mount Canigou, in the Pyrenees, at a height of 2785 metres. He then finds that the proportion $\frac{C}{H}$, i.e., of carbon to com-

bustible hydrogen, which was 3.49 in the air of Paris, and 2.2 in the air of woods, falls to 0.33 on the high mountain; thus showing that, in proportion as we rise in the air, away from the region of human industry and animal life, and also from the region of damp and vegetation, the hydrocarbons in the atmosphere tend to disappear.

The True Atomic Weights of Ten Elements deduced from Recent Work.—G. Hinrichs.—The results of various authors are discussed with regard to the atomic weights of hydrogen, carbon, oxygen, chlorine, bromine, sulphur, sodium, silver, barium, and boron.

A General Theory of Acidity.—M. de Forcrand.—The author's theory, at present very incomplete, appears to account for the greater quantity of organic and mineral compounds whose acidity is well known, from values which correspond in a remarkable manner with those given by experiment. The number of facts quoted is too great for the supposition of mere coincidence to hold good. When the theory is more fully developed, it will furnish a means of predicting the acidity of a hydrogen compound whose constitutional formula is known.

Hydrogenation of Acetylene and of Ethylene in presence of Platinum Black.—Paul Sabatier and J. B. Senderens.—The authors find that platinum black only induces the hydrogenation of ethylene when hot, whilst the hydrogenation of acetylene can take place in the cold after a considerable lapse of time.

Methoxyhydratropic Acid obtained by Oxidation of Anethol. Identity of Phloretic Acid and Hydroparacoumaric Acid.—J. Bougault.—In a preceding paper, the author showed that the oxidation of anethol

by iodine and yellow oxide of mercury gives an aldehyde of formula $C_{10}H_{12}O_2$, and that the oxidation of this aldehyde by silver oxide furnishes an acid of formula $C_{10}H_{12}O_3$ or $CH_3O-C_6H_4-C_2H_4-COOH$, and that this acid constitutes, with methylphloretic and methylhydro-paraconmaric acids, an isomeric series. Whilst examining the properties of these two latter acids, the author was struck by their similarity, and has since proved them to be identical.

Methods of Synthesising the Higher Homologues of Acetylacetic Ether and Acetylacetone.—L. Bouveault.—Method of formation of the compounds $C_2H_2(Cu_2Cl_2)_2KCl$ and $C_2H_2[(Cu_2Cl_2)_2KCl]_2$. This paper gives an account of the continuation of the work recently published on the transformation of yellow and colourless crystals corresponding to the above formulæ from one into the other.

Metallic Compounds of Diazoamidobenzene.—Louis Meunier.—The author prepares and examines the properties of the copper derivative of diazoamidobenzene, and also of the chlorohydrate, bromohydrate, and iodohydrate of the same derivative.

Action of Nitric Acid on Trichlorogaiacol.—H. Cousin.—The author finds that nitric acid acts on trichlorogaiacol, giving a body which is at the same time a product of oxidation and of condensation.

The Aloines.—E. Léger.—An account of the various derivatives of the aloines, their mode of preparation and their properties, are given in this paper.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xliii., No. 5.

Mathematical Laws of Chemical Equilibria.—O. Boudonard.—Already noticed in this column.

New Absorber-washer for Gas.—A. Gautier.—This absorber is made entirely of glass, and consists essentially of two bulbs of as near as possible the same capacity—that is, about 10 c.c.—connected by means of a wide vertical tube 10 or 12 c.m. in length, and also by a narrow bore serpentine tube surrounding the first-named one. At its lower extremity the central tube penetrates the lower bulb, to which it is fused, the end nearly reaching to the bottom of the bulb. The small serpentine tube starts from the middle of the side of the lower bulb, passes round and round the central tube, and joins the centre of the side of the upper bulb, to which it is fused; this latter terminates in a tube closed with a ground-glass cap and a side tube serving for the escape of the gas. To use this apparatus, the lower bulb is completely filled with the absorbent liquid (sulphuric acid mixed with anhydride, for example, if ethylene is to be absorbed), and the gaseous mixture is passed into the bulb through another tube joined into its upper side. The gas entering by this tube gradually fills the upper part of the bulb, forcing the liquid through the serpentine tube, by which it passes to the upper bulb, whence it again falls into the lower one through the central tube. At a given moment, however, the gas level reaches the level of the serpentine, and then, owing to a slight contraction in this tube which checks the flow, passes in bubbles up the serpentine. Arriving at the upper end of this tube, the gas escapes into the upper bulb, while the partly used liquid passes to the lower part of the lower bulb. In this manner the following triple result is attained:—(1) The gas is washed in the state of as small bubbles as may be desired; (2) the absorbent liquid is constantly being circulated and renewed; (3) The gas is washed with a minimum amount of absorbent liquid. With this apparatus, containing only 8 c.c. of potash of $d=1.3$, we have washed and absorbed all the carbonic acid from 200 litres of air.

Analysis of Water from Jouhe.—P. Bourcet.—Already inserted.

Remarks on the Cryoscopy of Rhamnose and Rhamnitrionic Acid of M. Tanret.—M. Ponsot.—The recent measurements made by M. Tanret on rhamnose and rhamnitrionic acid furnish us with two fresh examples of a coefficient of falling temperature very variable with the concentration. In most chemical work, when cryoscopy is used to decide between two figures for a molecular weight, one determination only is made, or the results of a single determination is given with a fall of about 1° ; this, the author thinks, is quite illusory.

Sulphides of Molybdenum.—M. Guichard.—Already noticed in this column.

Production of Crystallised Salts of Bismuth.—A. de Schulten.

Production of Iodised Carnallites of Potassium and Ammonium.—A. de Schulten.

Production of Vanadinites of Cadmium.—A. de Schulten.—These three articles have appeared in full.

Synthesis of Derivatives of Cyclopentane by the Use of Adipate of Ethyl. (II). Total Synthesis of the Pherone of Camphoric Acid.—L. Bouveault.—Already noticed.

Camphenylone.—E. E. Blaise and G. Blanc.—Already noticed.

Dinitration of Safranin.—G. F. Jaubert.—Already noticed.

Modifications undergone by Essence of Lavender during Vegetation.—E. Charabot.—The author finds that, as in the case of essence of bergamot, etherification is accompanied by a diminution of the total amount of alcohol and of free acids. These facts allow of the conclusion that here again the ethers take rise from the direct action of the acids on the alcohols. Under these conditions, during the development of the plant, one part of the linalol is etherified, while another part of this terpenic alcohol becomes dehydrated. On the other hand, as soon as the etherification is finished—which takes place when the plant is on its decline—we notice a fairly considerable increase in the richness of the essence in alcohol.

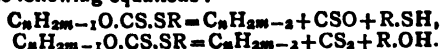
Remarks on the Metamorphoses and the Migrations of the Compounds of the Linalol Group in Plants.—E. Charabot.—The author has shown that linalol is first transformed during the growth of plants partly into ether and partly into terpenes. Essence of orange leaves contain a considerable proportion of acetic ethers of linalol and geraniol (about 60 per cent), as well as free linalol and geraniol (20 to 25 per cent). In the flower the proportion of geraniol with regard to linalol seems to have increased. When finally the essence becomes localised in the orange skin, the proportion of limonene becomes considerable, while the alcohols have almost entirely disappeared; the linalol has given limonene by dehydration in the parts where chloro-vaporisation takes place actively, and the geraniol is converted into citral in the organs which energetically fix oxygen.

MISCELLANEOUS.

Institute of Chemistry Examinations.—Pass Lists (July, 1900).—*Intermediate Examination*: J. A. Brown, Univ. Coll., Nottingham; E. M. Chapman, Pharmaceutical Society's Laboratories, and King's Coll., London; F. Cunliffe, Owens Coll., Manchester; J. A. Dewhurst, Yorkshire Coll., Leeds, and Pharmaceutical Society's Laboratories; W. D. Dick, King's Coll., London; A. W. Ellis, Mason Univ. Coll., Birmingham; A. Gray, Glasgow and W. of Scotland Tech. Coll.; J. E. Jenkins, King's Coll., London; W. Lowson, Yorkshire Coll., Leeds; W. W. Lumsden, Glasgow and W. of Scotland Tech. Coll.; B. G. McLellan, Glasgow and W. of Scot.

land Tech. Coll.; W. H. Nuttall, Univ. Coll., Nottingham; W. Partridge, Finsbury Tech. Coll., London; S. O. Richmond, Finsbury Tech. Coll., London; A. J. Robertson, Univ. Coll., Dundee, and under R. R. Tatlock, Esq., F.I.C.; P. W. Tainsh, Glasgow and W. of Scotland Tech. Coll.; W. S. Tebb, M.A., M.D. (Cantab.), Cambridge University, and King's Coll., London; J. Thorburn, Glasgow and W. of Scotland Tech. Coll.; F. W. Watson, Glasgow and W. of Scotland Tech. Coll. *General Practical Examination*: *A. Scott, Glasgow Univ., Glasgow and W. of Scotland Tech. Coll., and under R. R. Tatlock, Esq., F.I.C. *Final A.I.C. Examination*:—In *Mineral Chemistry*: A. Baguley, B.Sc. (Wales), Univ. Coll., Bangor; A. Davidson, Glasgow and W. of Scotland Tech. Coll.; *T. Hartley, Yorkshire Coll., Leeds; H. W. Kinnersley, Merchant Venturers' Tech. Coll., Bristol, King's Coll., London, and under E. H. Cook, Esq., D.Sc. (Lond.), F.I.C.; O. Reinherz, B.A. (Cantab.), Trinity Coll., Cambridge. In *Metallurgical Chemistry*: A. G. Levy, Finsbury Tech. Coll., London; *J. J. Morgan, of Wednesbury. In *Physical Chemistry*: T. H. Pope, A.C.G.I., City and Guilds of London Institute, Central Institution, and Finsbury Tech. Coll., London. In *Organic Chemistry*: F. G. H. Billows, A.C.G.I., City and Guilds of London Institute, Central Institution, and Finsbury Tech. Coll., London. In *the Analysis of Food and Drugs, including an Examination in Therapeutics, Pharmacology, and Microscopy*: S. Aston, Univ. Coll., London; N. P. Booth, Mason Univ. Coll., Birmingham; *T. W. Glass, B.Sc. (Lond.), Pharmaceutical Society's Laboratories, and under Messrs. T. H. Redwood and A. J. de Hailes, F.F.I.C.; W. H. Jollyman, Finsbury Tech. Coll.; *W. N. Platt, B.Sc. (Lond.), A.R.C.Sc. (Lond.), Royal Coll. of Science, London; *S. A. Woodhead, B.Sc. (Dun.), Durham College of Science, Newcastle-on-Tyne. The Examiners in Chemistry were Dr. Bernard Dyer, D.Sc. (Lond.), F.I.C., and Dr. W. Palmer Wynne, D.Sc. (Lond.), F.R.S., F.I.C. The Examiner in Therapeutics, Pharmacology, and Microscopy was Dr. Thomas Stevenson, M.D. (Lond.), F.R.C.P., V.-P.I.C. — (*For the Fellowship, F.I.C.).

A New Method for the Preparation of Non-saturated Hydrocarbides.—L. Techuagoff.—This new method rests on a splitting-up undergone by certain xanthogenic derivatives when submitted to dry distillation. If, for example, we distil the xanthogenic ethers of the general formula $C_nH_{2m-1}O.CS.SR$, where R represents an alcoholic radicle, a decomposition takes place according to the following equations:—



In both cases a non-saturated hydrocarbon is formed which it is easy to rid of the accessory sulphur products by distillation over metallic sodium. The transformation takes place at a relatively low temperature, and the returns are fairly satisfactory. We can in the same manner utilise the dixanthogenic compounds, which decompose according to the following equation:—



But in this case the formation of saturated hydrocarbides is much less satisfactory than it was previously. To transform menthol into menthane we add 16 grms. of sodium, in small quantities at a time, to a boiling solution of 100 grms. of menthol in 60–70 grms. of dry toluene, and heat for twenty hours on a sand-bath. After having separated the excess of liquid sodium we add the necessary quantity of CS_2 , and then treat the cooled solution with ICH_3 . In this manner we obtain methyl ether and methylxanthogenic acid, which crystallises in beautiful needles, fusible at 39° . When submitted to dry distillation, this compound decomposes, giving menthane and methylmercaptan as principal products. The boiling-point of the menthane is $167-168^\circ$; D_4^{20} : 0.8131, 0.8128,

0.8136. As for its rotatory power, the author found by two observations: $[\alpha]_D = +114.77^\circ$ and $+116.06^\circ$; that is to say, figures which are much more constant and much higher than those which have been given in chemical bibliography up to the present.—*Berichte*, vol. xxxii., p. 3332.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Asbestos.—Can any of your readers tell me where I can find a good account of asbestos and its physical structure? The works I am acquainted with give too meagre an account to be of any use.—**GEOFFREY MARTIN.**

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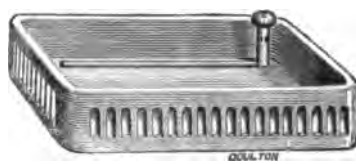
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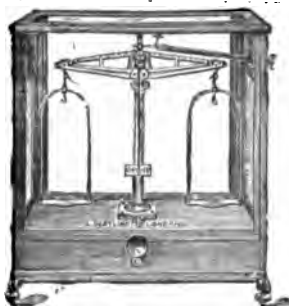
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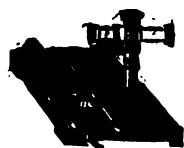
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VOL. LXXXII., No. 2124.

ON ELECTRIC TOUCH AND THE MOLECULAR CHANGES PRODUCED IN MATTER BY ELECTRIC WAVES.*

By JAGADIS CHUNDER BOSE, M.A., D.Sc.,
Professor of Physical Science, Presidency College, Calcutta
(Continued from p. 51).

[Electrical Reversal in the Radiation Product.]

In the hypotheses given above, it was said that the reaction of the radiation product, or B variety, should be opposite to that of the substance in the normal condition, or in the A state. Thus a negative substance which by the action of radiation shows an increase of resistance during conversion from the A to the B state should exhibit a diminution of resistance when B variety is acted on by electric waves. The contrary would be the case with positive substances.

The following tabulated statement indicates the phenomena exhibited by two classes of substances:—

Sign of electric touch.	Action of radiation on the fresh or A variety of the substance.	Further action of radiation on the radiation product or B variety.
Positive, e.g., iron	Diminution of resistance.	Increase of resistance.
Negative, e.g., arsenic	Increase of resistance.	Diminution of resistance.

We have thus two distinct classes of phenomena dependent on the sign of electric touch. If K_A represents the conductivity of the fresh substance, and K_B the conductivity of the radiation product, then—

(1). With positive substances, as the conductivity of the radiation product is greater ($K_B > K_A$), the first action of radiation would be to produce a diminution of resistance. This diminution will continue to be exhibited till the maximum amount of B variety is produced. The further action of radiation now will be to re-convert B into A; but as $K_A < K_B$ there would now be produced a diminution of conductivity, and a galvanometer in circuit will indicate an electrical reversal. The re-converted A variety may again be transformed to a greater or less extent to B, and in this way a series of reversals may take place, due to the continued action of radiation producing oscillation in molecular or atomic groupings. I shall designate this as the phenomenon of radio-molecular oscillation.

(2). With negative substances the conductivity of the radiation product is less ($K_B < K_A$), and the first action of radiation will therefore be an increase of resistance. The phenomena exhibited by these negative substances will precisely be opposite to those shown by the positive substances.

The above is but an approximate representation of the phenomena. To be more accurate, one has to take into account the partial changes and the effect of radiation on these changed products. Thus, at first suppose the substance to be entirely made up of A variety (this would rarely be the case). The first flash of radiation converts a large portion of A into B, the substance now being a mixture of A and B. The action of the next flash would be to convert the unchanged A into B, and re-convert to a more or less extent B into A. The electric response will thus be very strong at the beginning, but will become continuously less and less. When the proportion of B has attained a maximum value, the re-conversion of B into

A will become relatively large, and thus give rise to reversal effect.

I spoke of the conversion "to a greater or less extent" of one variety into the other. There is also the question of the relative stability of the two varieties under the given conditions. From the above it will be seen what possibilities there are in the way of different combinations, and the varied phenomena thereby rendered possible. I will presently describe some of the typical cases.

In the above it has been assumed that the reaction of B variety is opposite to that of A. As previously mentioned, in working with a silver receiver I found it, when fresh, exhibiting at first a diminution and, subsequently, an increase of resistance. The anomalous action may be explained by supposing the normal fresh silver Ag to be positive, and the radiation product Ag' negative. These two varieties would thus give rise to opposite reactions. To justify the assumptions made above, it became necessary to obtain by some other means a variety of silver Ag', analogous to the hypothetical negative variety].

Two Varieties of Silver.

After many unsuccessful attempts, I at last obtained a variety of silver which gives a moderately negative reaction (increase of resistance). Silver chloride was first precipitated by the addition of dilute hydrochloric acid to silver nitrate solution. The precipitate was then reduced to metallic silver by zinc filings, the excess of zinc being dissolved off by the action of HCl. This form of silver gives a negative reaction. Direct precipitation of silver produced by dipping a piece of zinc in AgNO_3 solution gives a positive variety. The negative product Ag' is perhaps better formed at relatively low temperatures, for the products obtained on certain warm days, the thermometer registering 27°C ., were very feebly negative, and passed into the positive state after an interval of twenty-four hours. But on cold days (temperature -22°C .) the products obtained were stable. I have specimens which have kept the negative property unimpaired for nearly three months. The negative property is not due to any accidental impurity, for pure silver obtained by Stas' method also gave the negative reaction. The negative reaction may, however, be supposed to be due to a thin film of chloride formed during reduction. I washed the Ag' with NH_3 , then with water, and, after drying, the result was still a negative reaction. I then carried out a parallel set of experiments with ordinary silver filings. Two separate quantities were taken; one was shaken with only HCl, the other was mixed with zinc filings, and the excess of zinc was dissolved off with HCl; the two specimens were then washed and dried. Both gave the positive reaction of ordinary silver. The above experiments are interesting for the production, by chemical means, of an allotropic variety analogous to the transitory radiation product.

There are other differences of electric behaviour between Ag and Ag'; for instance, when made into a voltaic cell, the two varieties give a P.D. of about 0.12 volt. There are other interesting peculiarities about this cell, the consideration of which is postponed to a future occasion.

Electric Reversal.

It now remains to be proved that the "radiation product" exhibits a change of sign of electric touch. The sensitiveness of certain substances belonging to each of these two classes is very great. On the other hand, in the transition from one class to the other, substances are met with the sensitiveness of which is rather feeble. The experimental verification of the hypotheses mentioned above seemed at first very difficult, as the reversed action was likely to be masked by the stronger normal action of the still unconverted portion of the substance. It however occurred to me that if slightly sensitive substances were taken, then the direct and reversed actions were likely to be obtained with less difficulty. For this reason I took for my first experiments arsenic, which is moderately

* A Paper read before the Royal Society, February 8, 1900.

negative. It is, however, possible, though the adjustments are difficult, to exhibit the reversed actions even with strongly sensitive substances, and as a type of such actions that of iron will be taken as an example.

Observations with Arsenic Receiver.—*Experiment I.*—A receiver was made with freshly powdered arsenic; the critical distance was found to be 25 c.m.; that is to say, when the radiator was moved from 1 to 25 c.m. there was always produced an increase of resistance, while beyond this distance there was a diminution of resistance; the critical distance 25 c.m., may therefore be taken as an approximate measure of the negative character of the specimen. As has been said before, if through any cause the substance becomes more negative, the critical distance will be increased; but if the substance tends towards the positive direction by becoming less negative, then the critical distance will be decreased. The receiver was now continuously subjected to radiation for ten minutes. After this it was found that the receiver gave a diminution or positive reaction, even when the radiator was brought

turbance was found to break up the complex atomic aggregation in the radiation product. During eight minutes of exposure the receiver continued to exhibit an increase of resistance, after which the substance became positive, being converted to the B state; this positive state lasted for a minute under exposure to radiation, then there was a reversal to the original negative state—as if the structure so laboriously built up suddenly gave way. Subsequently there were series of reversals, the specimen becoming more and more inert, and, after an exposure of about thirty minutes, the sensitiveness was practically lost.

The curve given below (Fig. 1) represents approximately the results of the experiment. During certain periods the substance became so nearly neutral that it was difficult to interpret whether the substance was positive or negative. The lower halves of the curve represent the negative, and the upper halves the positive states, and the corresponding numbers represent, in minutes, the duration of these states.

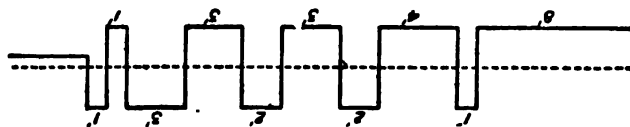


FIG. 1.—ELECTRIC REVERSAL CURVE.

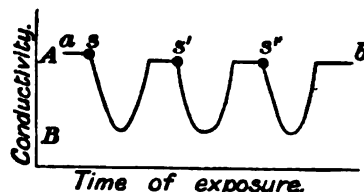


FIG. 2.—CURVE FOR POTASSIUM.

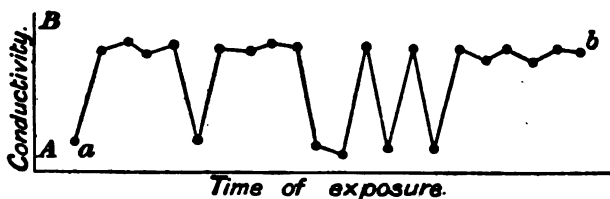


FIG. 3.—RADIO-MOLECULAR OSCILLATION CURVE FOR MAGNESIUM.

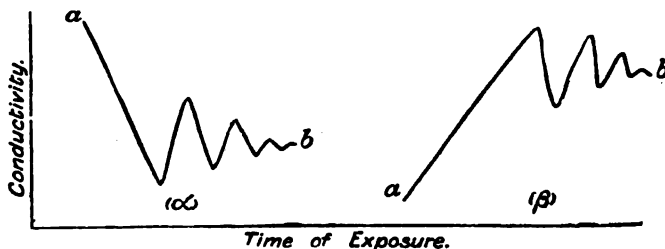


FIG. 4.—“DAMPED” MOLECULAR OSCILLATION.

close to the receiver. The action of radiation has thus reversed the sign of electric touch.

Experiment II.—Any arbitrary length of exposure labours under the defect that what is observed is the final effect, the intermediate effects not being taken into account. In order to observe the intermediate effects, a very laborious series of observations is necessary. The experiment was therefore modified in the following manner:—A fresh receiver was subjected to radiation, and observations at intervals of fifteen seconds were taken to test the nature of reaction of the sensitive substance. The first action of radiation on the fresh specimen was a great increase of resistance—so very great that the current was reduced to zero; it was no longer possible to make any further observation without re-establishing the current. This was done by a very gradual increase of pressure, effected by means of a fine micrometer screw which moved the compressing electrode in a perfectly parallel manner. There should be no jarring motion, as mechanical dis-

[Radio-molecular Oscillation.

It was said that, owing to molecular reversals due to radiation, there should be a corresponding series of electric reversals. In this investigation it is essential that the substance examined should be completely protected from all disturbances, such as mechanical vibration, &c., and only subjected to the action of radiation. The experimental difficulties are very great. If we take a strongly sensitive positive substance—say iron, the effect of the first flash of radiation (a diminution of resistance) is very great, and the subsequent relatively feeble reversal effects, unless carefully looked for, are liable to pass unnoticed. After the first adjustment, the receiver should on no account be touched, as mechanical jars are found to undo the effect of radiation. Though by special care the mechanical jars could be reduced to a minimum, yet it appeared advisable to devise appropriate means by which the necessity of touching the receiver for subsequent adjustments is altogether avoided. The method

adopted to this end varies with individual cases. In the case of arsenic, for example, the action of radiation is often to produce a very great increase of resistance, and thus convert the substance into a non-conductor. The pressure has therefore to be so adjusted at the beginning, that the substance can never become altogether non-conducting; the receiver is thus absolutely free from all effects, except those which are to be observed—viz., the effects due to continuous action of radiation. The time of exposure is accurately measured by counting the individual flashes of radiation due to interruption of the primary current in the Ruhmkorff coil by a tapping key. The conductivity of the substance at a given moment is inferred from the deflection of the galvanometer in circuit with the receiver. When feeble radiation is used, it takes an inconveniently long time to obtain reversals; there is, besides, a tendency of self-recovery in the receiver. In order to expedite the reversals, the incident electric radiation is made very strong.

Before entering into the detail of the results obtained, I will say a few words about the principal types likely to be met with. We may have the following:—

I. Substances in which the B state is unstable under the given conditions; the B state will therefore only persist during the action of radiation, the substance relapsing into the original condition on the cessation of radiation. Two cases are possible (i.) when the substance is positive, (ii.) when the substance is negative.

The latter case is exemplified by potassium. In the curve (Fig. 2), A and B represent the two molecular states. The substance being negative, A is more conducting; *a* represents the conductivity of the fresh specimen; the thick dots S S' . . . the individual flashes. It is seen that the effect of radiation is to produce a sudden diminution of conductivity, due to the transient formation of B variety with its diminished conductivity. The substance, electrically speaking, is highly elastic, and the limit of its elasticity is also very great. With the majority of substances, however, self-recovery is only possible when the narrow limit of elasticity is not exceeded.

II. In this class the radiation product is somewhat stable; the successive conversions from A to B and from B back to A are supposed to be complete. Probably there is no substance which exhibits this action in a perfect manner, but we have an approximation to this condition in the case of magnesium, which under proper adjustments shows successive complete reversals for a long time. The substance, however, after a time exhibits the effect of fatigue.

The curve given in Fig. 3 clearly exhibits the reversals. This curve also explains the behaviour of magnesium noticed in my last paper, which appeared very curious at the time. "It is sometimes possible to so adjust matters that one flash of radiation produces a diminution of resistance, and the very next flash an increase of resistance. Thus a series of flashes may be made to produce alternate throws of the galvanometer needle" ("On a Self-recovering Coherer," &c., *Proc. Roy. Soc.*, vol. lxx., p. 169).

The receiver was so adjusted as to give a deflection of five divisions.

The first flash of radiation produced an increased deflection of 50 divisions (magnesium having a positive electric touch); the second flash produced a further deflection of five divisions, the third flash produced a *negative* deflection of five divisions, the fourth flash produced + 5, the fifth flash gave - 90 divisions, and the sixth flash a deflection of + 90. The reversals followed each other almost regularly, till the substance became insensitive.

III. Lastly, we may have a class of substances where the conversion from one state to the other is not complete. Here, again, we get two sub-divisions, owing to the distinction between positive and negative substances.

Taking first the case of a positive substance (see Fig. 4, B), the original conductivity of which is represented by *a*, the action of the first few flashes of radiation would be to produce a great increase of conductivity by the

formation of B variety; the next flashes convert B back to A, but not completely, and the negative deflection will be less than the previous positive deflection. Owing to this "damping" effect, the oscillation curve will approximate to a logarithmic decrement curve. After a series of

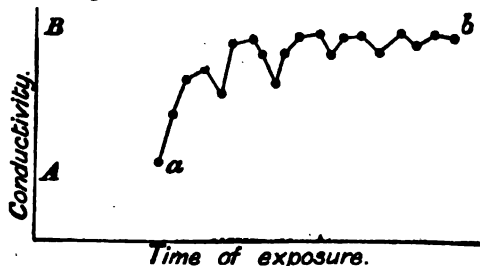


FIG. 5.—"DAMPED" OSCILLATION CURVE FOR A POSITIVE SUBSTANCE.

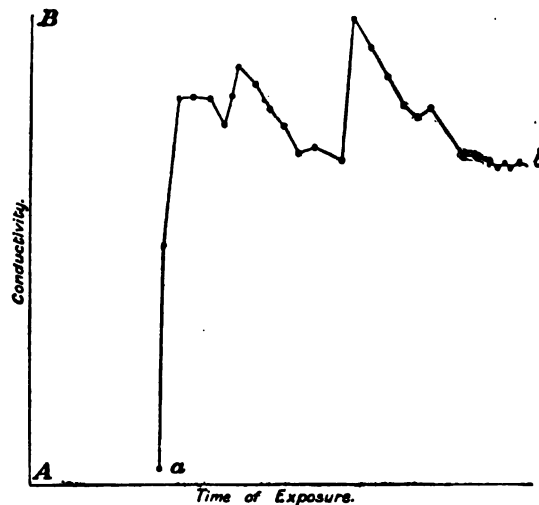


FIG. 6.—CURVE FOR IRON.

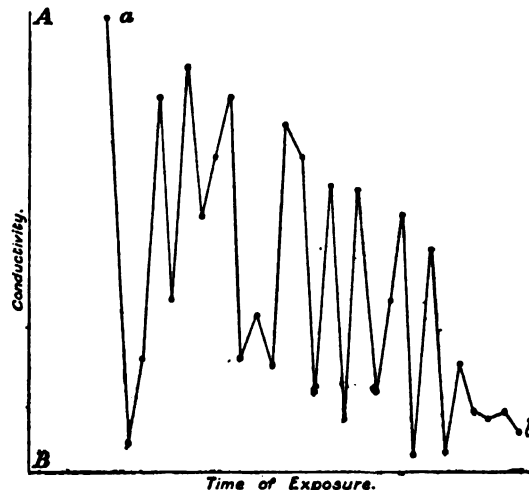


FIG. 7.—CURVE FOR ARSENIC.

reversals the oscillation dies away, and the substance becomes almost inert. A glance at the hypothetical curve to the right shows that at the inert stage, *b*, the substance as a whole ought to become more conducting than the fresh specimen, *a*.

The opposite should be the case with negative substances (see Fig. 4, a).

Fig. 5 exhibits the actual curve obtained with a compound positive substance.

It is remarkable for its regularity. The next figure (Fig. 6) gives the curve for iron. The first diminution of resistance is too great to be properly represented in the diagram. Here we have the same type as in the previous case; *the inert stage, b, is also more conducting than a.*

IV. I will now consider the case of a negative substance exhibiting damping; arsenic will be taken as an example where the damping is not so great as in the case of iron.

Fig. 7 represents the actual curve obtained with arsenic (compare with the hypothetical curve for a negative substance, Fig. 4, a).

It will be observed that *the substance in the fatigued state is, on the whole, less conducting than in the fresh condition*, as we are led to expect from the hypothetical curve. It will also be seen that the oscillations are very regular towards the end. The curves given in Figs. 6 and 7 are those obtained with specimens immediately after they were set up. Had I allowed them a period of rest to allow the particles to get properly settled, I would have got curves even more regular. It is, however, evident that in substances exhibiting damping, two opposite electric conditions are induced in fatigued specimens; the positive becomes on the whole more conducting and the negative less conducting than in the fresh specimens. At the inert stage the rate of mutual conversion from one state to the other probably becomes equal, and the apparent fatigue is thus not due to the absolute want of sensitiveness of the constituent varieties, but to the opposite reactions of A and B balancing each other.

We may now apply some further crucial tests to verify the suppositions made above.

(To be continued).

ON THE COMPOSITION OF N. S. WALES LABRADORITE AND TOPAZES, WITH A COMPARISON OF METHODS FOR THE ESTIMATION OF FLUORINE.*

By G. HARKER, B.Sc.

(Concluded from p. 57).

Results obtained by the Tetrafluoride Method on Calcium Fluoride, Cryolite, and Apatite.—First Series.—The substance was fused with sodium potassium carbonate and then decomposed. The calcium fluoride used in these experiments was from ground crystals, and on testing, by conversion into calcium sulphate, gave 99.6 per cent of calcium fluoride. Four experiments gave 44.60, 45.97, 46.12, and 46.20 per cent, the theoretical percentage of fluorine being 48.53 and the average difference 2.82 per cent.

Second Series.—The substance was decomposed without previous fusion with alkaline carbonates. The calcium fluoride in the first four of these experiments was from crystals of fluor-spar ground, and evaporated to dryness with hydrofluoric acid to get rid of silica, and then ignited. On testing by conversion into calcium sulphate they gave 99.8 per cent of calcium fluoride.

	Amount taken. Gms.	Percentage of fluorine found.
1	0.1565	45.90
2	0.2722	47.08
3	0.5212	47.21
4	0.2900	46.64
5	0.1736	46.31
6	0.1787	45.95

The theoretical percentage was 48.62 and the average difference 2.25 per cent. These results were corrected for the solubility of potassium silicon fluoride, which was found to be 0.0016 gm. in 50 c.c. of a 50 per cent alcohol solution. Nos. 5 and 6 were precipitates of calcium fluoride obtained by Berzelius's method from the Mudgee topaz.

Cryolite similarly gave 52.17 per cent when previously fused with alkaline carbonates, and 52.62 without previous fusion, and when treated by Berzelius's method 55.34. Apatite gave 2.80 by the first method and 2.81 by the second.

Application of the Tetrafluoride Method to Topaz.—The Mudgee topaz by this method gave only 13 per cent of fluorine, which was much too low. When repeated on the Brazilian topaz a similar low result was obtained, viz., 10.9 per cent. It seems, therefore, that this method works well for minerals not containing silica. That it is the presence of silica in the topaz which causes the error was shown by adding silica to calcium fluoride or topaz before fusing, when in all cases a much lower result was obtained, the error being larger as more silica was added. Thus calcium fluoride gave 36.54 and 35.06 per cent of fluorine, and topaz 7.05 and 4.26 per cent, results much lower than those obtained when silica was not added.

Third Method, Fusion with Alkaline Carbonates and Silica.—A third method for the determination of fluorine was then tried, viz., that of Berzelius as modified by Rose. The ground topaz is fused with one-half its weight of pure silica; the fused mass extracted with water; 5 to 10 grms. of ammonium carbonate added to precipitate silica, and the solution allowed to stand twelve hours. The solution is filtered and evaporated to get rid of excess of ammonia, and a solution of zinc carbonate in ammonia added to precipitate any silica in solution. The liquid is then further evaporated to get rid of ammonia, filtered from zinc oxide and silicate, and the fluorine precipitated by calcium chloride in the usual manner.

The results from the Mudgee topaz were much higher than those obtained by the two previous methods, and were constant; the same constant results were obtained in the case of the Brazilian topaz and the green topaz from New England.

The calcium fluoride obtained by precipitation in this method from the Mudgee topaz was tested and found to be free from calcium silicate. Berzelius's method therefore gave the best results for fluorine in topaz; but as there seemed to be no reason why the fluorine should be lost in Wöhler's method, which did not apply equally to that of Berzelius's, an attempt was made to find where the fluorine was lost in the former case.

It was found that no fluorine is left in the residue after extracting the fused mass with water. The decomposition in Wöhler's method is therefore complete. It was noticed that the precipitate of aluminium hydroxide obtained on addition of ammonium carbonate to the solution was more granular than the usual gelatinous precipitate, and on trial this precipitate was found to contain no silica, but always more or less fluorine. (Hydrofluoric acid was liberated from the precipitate by sulphuric acid and etched glass. Mixed with silica and sulphuric acid, silicon tetrafluoride was given off, which was passed into water and the potassium silicon fluoride precipitated). On one occasion 6.6 per cent of fluorine was obtained in this precipitate by Berzelius's method, but this was exceptionally large, the usual amount not being more than 1 to 3 per cent. When the ammonium carbonate was added hot to the solution, and then allowed to stand until quite cold, a larger proportion of fluorine was found in the precipitate than when it was filtered hot, the precipitate containing the fluorine coming down on cooling. This precipitate contained a larger amount of fluorine when sodium potassium carbonate was used than when sodium carbonate alone was employed. In obtaining the results given by Wöhler's method for fluorine, sodium potassium carbonate was frequently employed, which accounts for

* Read before the Royal Society of N. S. Wales, December 6, 1899.

some of them being so low, but, even when sodium carbonate alone was employed, the highest percentage obtained was 12.69.

On adding the amount of fluorine recovered from the precipitate to the amount obtained from the solution after the addition of ammonium carbonate, the total is nearly the same as that obtained by Berzelius's method. Thus 12.2 per cent was obtained from Brazilian topaz by Berzelius's method. In one experiment 6.92 per cent was obtained from the solution and 6.50 from the precipitate, total 13.42 per cent. In a second experiment 12.69 from the solution and 1.21 from the precipitate, total 13.90 per cent. This seems to show that Berzelius's method gives the total fluorine present. The precipitates of calcium fluoride were tested by conversion into calcium sulphate. The residue obtained on treating the fused mass with water in Wöhler's method contained all the silica and about two-thirds of the alumina and some soda, and about approximately the constitution $3\text{SiO}_2, 2\text{Na}_2\text{O}, 2\text{Al}_2\text{O}_3$. On boiling with water the soda and alumina were slowly removed, but to what extent this could be carried out was not determined. In the case of Berzelius's method the residue contained all the aluminium. The silica prevents the aluminium from passing into solution, and thus stops the loss of fluorine which occurs in Wöhler's method. The amount of silica necessary to do this was found to be 31.6 per cent in the case of the Brazilian topaz.

Examination of the Ammonium Carbonate Precipitate.—The topaz was fused with sodium carbonate alone and the fused mass dissolved in water and filtered. Ammonium carbonate was added to the hot solution, and a gelatinous precipitate, consisting chiefly of aluminium hydroxide, formed. After filtering and cooling a more granular but still gelatinous precipitate separated out and collected at the bottom of the beaker. This was filtered off and examined. Only a small amount was obtained, which on drying became powdery and fused at a red heat. The sodium and aluminium were determined by conversion into sulphates, the fluorine by fusion with silica and alkaline carbonates. 0.0210 grm. heated to redness gave 23.8 per cent sodium, 26.2 per cent aluminium; and 0.200 grm. gave 33.3 per cent of fluorine, leaving oxygen by difference 16.7 per cent. Assuming some of the aluminium to be combined with the oxygen as alumina due to imperfect separation of the fluorine precipitate from aluminium hydroxide, we have for the composition of the former fluorine, precipitate sodium 36.8, aluminium 11.7, and fluorine 51.5 per cent. Cryolite requires 32.9, 12.8, and 54.3 per cent.

In another experiment on a different precipitate 0.0130 grm. gave sodium 20.8, aluminium 29.2 per cent, and 0.201 grm. gave 30.8 per cent of fluorine, hence oxygen by difference 19.2 per cent. Allowing for alumina this gives sodium 35.1, aluminium 13.0, fluorine 51.9 per cent.

It seems probable therefore that the fluorine is precipitated as a double fluoride of sodium and aluminium. Like cryolite, this precipitate also fuses readily. But as the amounts analysed were so small, it is proposed to confirm the results by working with larger quantities.

Comparison of the various Methods for Fluorine on the Topazes.

	1. Fusion with alkaline carbonates.	2. Tetra- fluoride.	3. Fusion with alkaline carbonates and silica.
N. S. W. topaz from Mudgee	5, 11.01	12.93	17.04, 17.60, 17.10
Brazil	4.73, 11.2, 11.71, 11.21, 6.92, 12.69	10.90	14.23, 14.61
New England topaz	—	—	16.30, 15.92

A table is given above showing the results obtained by the various methods for fluorine, on the topazes. While obtaining the percentages for fluorine by methods (1) and (3), the silica and alumina were also estimated, so

that, with the exception of water, the whole of the constituents were now obtained.

It has been found by Penfield and Minor (*Am. J. Sc.*, 94) and Jannasch and Locke (*J. Chem. Soc.*, 94) that all topazes contain more or less water of constitution. The former found 2.45 and the latter 2.69 per cent in Brazilian topaz. The water takes the place of the fluorine. Penfield and Minor decomposed the topaz with sodium carbonate, and the water free from acid was weighed in sulphuric acid or calcium chloride tubes. Jannasch and Locke decomposed with litharge in a bulb tube and collected the water in a calcium chloride tube, a stream of dry air being passed through the apparatus. This last method was found more convenient, as a much lower temperature could be used to decompose the topaz; both methods, however, gave the same results. In the experiments the topaz was first heated to bright redness over a strong Bunsen to drive out any contained water; it was then decomposed by litharge and its so-called water of constitution determined. Blank tests on the litharge showed that the calcium chloride tube gained 2 to 3 decim. grms., and in calculating the percentages this was deducted. In the experiments about 0.5 grm. of topaz was mixed with 2 to 2.5 grms. of previously heated litharge, the same amount of litharge being used in the blank tests.

Water of Constitution.

Locality.	Amount taken. Grm.	Water found.	Percentage.
Brazil	0.6324 0.5080	0.0134 0.0107	2.12 2.11
New England	0.5038 0.5780	0.0050 0.0066	0.99 1.14
Mudgee	0.4900 0.4958	0.0038 0.0038	0.78 0.75

Results of Analyses.—Appended is a table giving the complete analyses of these three varieties of topaz. In the latter it will be seen that the silica and alumina obtained by Wöhler's method are always higher than by Berzelius's, and this is apparently due to the aluminium hydroxide taking down with it some of the fluorine and sodium.

The alumina was always tested by potassium bisulphate, and the silica by hydrofluoric acid purified from the commercial acid by re-distilling in a platinum retort. The calcium fluoride precipitates were tested by conversion into calcium sulphate or in some other way.

Analysis of Topazes.

	Fusion with alkaline carbonates and silica.	Fusion with alkaline carbonates alone.		
	1. Mudgee, N. S. W.	2.	1.	2.
SiO ₂	31.90	31.84	32.30	32.46
Al ₂ O ₃	56.62	56.80	59.70	59.21
F	17.60	17.00		
H ₂ O on ignition	0.23	0.26		
H ₂ O by PbO ..	0.75	0.75		
	107.10	106.65		
	Brazil.			
SiO ₂	31.95	32.16	32.31	31.90
Al ₂ O ₃	54.52	54.61	55.90	56.45
F	14.62	14.23		
H ₂ O on ignition	0.23	0.30	0.26	0.24
H ₂ O by PbO ..	2.12	2.12		
Fe ₂ O ₃	—	—	—	0.10
	103.44	103.42		0.09
	New England, N. S. W.			
SiO ₂	31.73	31.92		
Al ₂ O ₃	55.62	55.43		
F	16.30	15.92		
H ₂ O on ignition	0.37	0.39		
H ₂ O by PbO ..	1.07	1.07		
Fe ₂ O ₃	0.12	—		
	105.21	104.73		

Analyses of Topazes from other Sources.

	CURRAM.	PENFIELD AND MINOR (<i>Am. Journ. Sci.</i> , 94).			LIVERSIDGE. Shoalhaven, N. S. W.*
		N. S. Wales.	Utah.	Stoneham. Brazil.	
SiO ₂ ..	30.29	31.93	32.28	32.53	28.19
Al ₂ O ₃ ..	60.90	56.25	56.33	55.67	62.66
F ..	15.05	20.33	18.56	15.48	14.01
H ₂ O ..	0.40	0.19	1.04	2.45	—
	106.64	108.71	108.17	106.13	104.86

* Dull and impure.

INTERNATIONAL COMMISSION ON ATOMIC WEIGHTS.

THE undersigned members of the International Commission on Atomic Weights welcomed the appeal from the German Chemical Society for a universal standard table of atomic weights, but have come to the conclusion that, starting from the basis O=16, proposed by Bohuslav Brauner in 1888, no unanimity can be reached. Many and daily are the voices urging grave reasons against the abandonment of the standard H=1 (*Cf.* Lassar-Cohn, "On the Unsuitability of the Recently Proposed Number 16.000, as the Basis for Reckoning Atomic Weights," *Hambourg, Leopold Voss*, 1900; and Böhm, "On the New Atomic Weights Table," *Chemiker Zeitung*, 1900, No. 47, p. 495).

If cogent reasons necessitate an alteration of the standard of our atomic weights, it would be better to start from an element whose weight can be conveniently ascertained, an element such as, e.g., silver or iodine, which also serves as a practical starting-point in consideration of the sharpness of its reactions in numerous analytical operations.

But, in our opinion, such cogent reasons for an alteration do not present themselves (*Cf.*, *Journal for Applied Chemistry*, 1899, pp. 424, 570, 648, 980—990; for 1900, pp. 376, 463). The ratio of hydrogen to oxygen has been established by the researches of Keiser, Scott, Rayleigh, Cooke, Richards, Noyes, Dittmar, Henderson, Leduc, Morley, Thomson, and Berthelot, with an exactness which fully suffices for all practical purposes.

The time for an unchangeable table of atomic weights has not yet come; each succeeding year brings corrections in the atomic weights of the rarer elements, and at the same time speculations as to their simple or compound nature.

For the teacher, simplicity and clearness of the foundation seem specially important; instruction must suffer no harm with regard to the enlightening construction of the law of volumes, no shadow of doubt must penetrate the doctrine of valency. Regard for the understanding of prospective chemists will compel us therefore, under all circumstances, in teaching, and in our text-books, to retain Dalton's numbers (Senbert, *Berichte*, 1898, xxi., p. 2776; Landolt, *Berichte*, 1898, xxxi., p. 2767), and Prof. F. W. Clarke, the worthy editor of the Annual Atomic Weight Tables of the American Chemical Society, authorises us to say that he recommends the retaining of the standard H=1. For if numbers were used in practice which were unsuitable to use in teaching, confusion would be the natural consequence, instead of the unanimity desired by all.

The Commission on Atomic Weights of the German Chemical Society intend by the publication of these opinions to give all chemists the opportunity of expressing themselves on the question of universal atomic weights. General requests of this kind do not as a rule secure the desired attention; but we consider it of the highest importance that teachers of chemistry in universities and technical high schools take up a definite position with

regard to this matter. We therefore beg to be allowed to lay before the readers of the *CHEMICAL NEWS* the following questions:—

1. Shall the unity of hydrogen be retained as the standard for reckoning atomic weights?
2. Shall the atomic weights be given approximately with two decimal places in which the uncertain figures can be recognised by the type?
3. Shall the International Atomic Weight Commission have the current table of atomic weights edited on this basis?

We beg our colleagues to express their opinions with regard to these questions, and to send their answers* at their earliest convenience to Herr Prof. J. VOLHARD, Halle-a-S., Mühlporte 1.

J. BREDT,
H. ERDMANN,
FERD. FISCHER,
J. VOLHARD,
CL. WINKLER,
J. WISLICENUS.

Halle-a-S., July 6, 1900.

THE ESTIMATION OF COBALT IN
NEW CALEDONIAN ORES.

By THOMAS MOORE, Nouméa.

THE ores in question consist mainly of hydrated oxides of manganese, iron, alumina, cobalt, and nickel; the other constituents—such as lime, magnesia, zinc oxide, lithia, &c.—are only in relatively small proportions. Barium and copper are only occasionally present. The cobalt varies from traces up to 8 per cent. The average may be taken as 5 per cent, whilst the proportion of nickel varies from one-third to one-half of the cobalt. The mineral is sold only according to its contents in cobalt reckoned as monoxide, no notice, so far as the miner is concerned, being taken of the other metals.

The principal difficulties encountered in the assay of the mineral are the complete separation of the cobalt from the iron oxide and alumina, and the estimation of the separated cobalt. The separation from the iron and alumina is generally carried out by the basic acetate method in England, a second precipitation being invariably resorted to; on the Continent the baric carbonate process appears to find most favour. The cobalt estimation is usually confined to two methods, deposition electrolytically as metal, or precipitation as ammonio-cobalto phosphate.

In the following process I have endeavoured to shorten and simplify the operations as much as possible, and at the same time to obtain satisfactory and concordant results:—

Two and a half grms. of the pulverised and dried mineral are dissolved in concentrated hydrochloric acid, and evaporated until the solution becomes syrupy; water is now added to dissolve all soluble matter, leaving only a residue of silica and chromite; 100 c.c. of a saturated solution of ammonic chloride are now added, and the whole then diluted with cold water to about 400 c.c. A weak solution of ammonia (1 part strong ammonia and 15 parts water) is now carefully added with constant and vigorous stirring, until the liquid takes a deep red colour, indicating the formation of basic ferric compounds. The solution must be clear and free from precipitated oxide of iron, which is easily distinguished from the turbidity caused by the silica in suspension. From this point the neutralisation is completed by the careful addition, drop by drop, of a 5 per cent solution of sodic carbonate until

* We invite correspondents to send their replies and comments to the *CHEMICAL NEWS* for publication.—Ed. C. N.

the supernatant fluid is perfectly clear and transparent. To arrive exactly at this condition it is preferable to stop the addition of the sodic carbonate when the solution has only a slight yellow tint, as a little extra stirring invariably causes this to disappear. At this stage the solution, which possesses a distinct acid reaction, but is nevertheless free from iron or alumina, is transferred to a half-litre flask and diluted to the mark; then, after a thorough mixing by shaking, filtered through a ribbed filter: 400 c.c. of the clear filtrate are now exactly measured off into a flask of about 600 c.c. capacity, 20 c.c. of a saturated solution of sodic acetate and 10 c.c. of acetic acid added, and the whole then raised nearly to the boiling-point. A current of sulphuretted hydrogen is then passed through the solution, allowing the gas to pass until the contents of the flask are nearly cold; the sulphides of cobalt, nickel, and zinc will then be found to have been completely precipitated free from impurities, and may now be filtered off, washed with water containing sulphuretted hydrogen, then dried and ignited. The ignited oxides and any sulphide which may remain attached to the sides of the flask are now dissolved in strong hydrochloric acid with the aid of a drop or two of nitric acid, and evaporated twice to dryness with hydrochloric acid, so as to completely get rid of all nitric acid, the residual chlorides dissolved in from 5 to 10 c.c. water, and allowed to become cold. The solution of the metals thus obtained may through careless neutralisation contain a little iron, so that for greater security it is always better to add a slight excess of an emulsion of oxide of zinc, and filter off any trace of ferric oxide which may be thrown down.

The filtrate, which is now entirely free from iron, is diluted to about 50 c.c., and well mixed with from 10 to 15 c.c. of a 10 per cent solution of hydrogen peroxide, and then with 10 c.c. of a 10 per cent solution of potassic or sodic hydrate; the cobalt is immediately precipitated as sesquioxide, Co_2O_3 , whilst the nickel is simply thrown down as the green hydrated monoxide. The mixed precipitate is now boiled for about a minute, so as to ensure the complete decomposition of the excess of hydrogen peroxide, allowed to cool, and then transferred to a stoppered bottle and digested with an excess of potassic iodide and hydrochloric acid until the decomposition is complete; then the iodine thus set free is titrated in the usual manner with sodic hyposulphite, $1 \times 0.46511 = \text{Co}$. The potassic or sodic hydrate employed in this process must be previously examined for impurities which can liberate iodine from potassic iodide; samples containing nitrites, for example, are not uncommon.

As illustrating the accuracy of this process, the following examples will show what may be expected:—

Two lots of ore, each of 2½ grms., were treated as described, A without any addition; whilst to B, 10 c.c. of a perfectly pure solution of cobalt, containing 0.0920 grm. of CoO , were added immediately after the mineral was dissolved.

	A.	B.	Difference.	Error.
1.	4.21	7.91	3.70	0.02 % CoO
2.	6.67	10.38	3.71	0.03 „
3.	6.84	10.53	3.69	0.01 „
4.	7.73	11.43	3.70	0.02 „

Essence of Chrysanthemum.—G. Perrier.—Essence of chrysanthemum is a greenish liquid of an oily consistency, with a special odour recalling both that of peppermint and camomile; it begins to boil at 160° , its density is 0.932 at 15° , and its index of refraction at 18° is 1.4931. It is soluble in ten parts of alcohol at 95° , but almost insoluble in alcohol at 70° . When cooled down to -15° it deposits a small quantity of an amorphous solid product, probably a paraffin; at -24° it becomes very thick, and solidifies completely in a mixture of ether and solid carbonic acid.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 6.

THE ANALYSIS OF CHROME AND TUNGSTEN STEELS.

By A. G. M'KENNA.

THE writer has had occasion during the past few years to make complete analyses on several hundred samples of steel, containing both chromium and tungsten, and has found the following methods very satisfactory, accurate results being obtained without excessive care or unusual precautions.

The steel is usually too hard to be drilled, and is broken up in a steel mortar until no single piece is larger than a grain of rice. For the determination of sulphur, silicon, tungsten, manganese, and chrome, 5 grains are weighed into a 500 c.c. evolution flask, so arranged that the gases evolved on the addition of 30 c.c. hot water and 30 c.c. concentrated hydrochloric acid shall be absorbed by an ammoniacal cadmium chloride solution contained in a large test-tube. The solution is made as rapidly as possible by the application of heat, as there seems to be evidence that with some irons lower results are obtained by the evolution method when cold acid is used. When the steel has dissolved the solution is boiled for a minute or two until no more hydrogen remains in the flask, being displaced by steam. The sulphur is then determined by titration with iodine in the usual manner, using starch solution as an indicator. By adding a few grains of zinc chloride to the starch solution it can be kept indefinitely without spoiling. The solution in the evolution flask is transferred to a 500 c.c. Erlenmeyer flask, 10 c.c. of strong HNO_3 added, and evaporated to dryness on a hot plate, taken up with 15 c.c. strong HCl , evaporated again to dryness, taken up in 20 c.c. strong HCl , diluted with hot water to about 100 c.c., boiled, and filtered. All the silica and tungstic acid will be on the filter-paper; after washing thoroughly with a 5 per cent HNO_3 solution, the residue is ignited in a weighed platinum crucible, $\text{WO}_3 + \text{SiO}_2$, and weighed. A few drops of HFl are now added, and the crucible is heated to a bright red for five minutes to volatilise silica. The loss is silica, which is calculated to silicon; the residue in the crucible is tungstic acid, which is calculated to tungsten. This residue generally contains a trace of iron oxide, which can be easily determined by fusing the residue, after weight has been taken, with sodium carbonate, and filtering off the oxide of iron after solution in hot water.

The filtrate from the tungstic acid and silica is again transferred to an Erlenmeyer flask and evaporated to low bulk; 50 c.c. of concentrated HNO_3 are now added, and the solution boiled until no more fumes come off, showing that all hydrochloric acid has been removed. Enough concentrated HNO_3 is added to make the volume 200 c.c., and the solution again heated. When it has reached the boiling-point, 10 grains of potassium chlorate are added, and the solution boiled down to 75 c.c. in order to remove all chlorine.

The manganese will now be completely precipitated as MnO_2 , and the chromium will be converted to chromic acid.

The solution is filtered on an asbestos plug while hot, and washed a few times with freshly boiled concentrated nitric acid. In the filtrate chromium is determined by titration with ferrous sulphate and permanganate according to the following reactions:—



If the solution is diluted to about 500 c.c., and cooled to about 20°C . before titration, there is not the slightest danger of interference by the nitric acid. The MnO_2 on the asbestos plug is dissolved by hot hydrochloric acid and a pinch of potassium nitrite. It is brought to a boil to drive off chlorine and the traces of iron precipitated by ammonia and ammonium acetate; the basic precipitate is dissolved and re-precipitated to free from traces of

manganese. In the filtrate manganese is precipitated by bromine in a strongly ammoniacal solution, filtered, ignited, and weighed as Mn_2O_3 .

For the determination of phosphorus 5 grms. is weighed into a porcelain dish, and 60 c.c. of dilute nitric acid added. If the steel contains more than 1 per cent of chromium, it will probably be found necessary to add HCl from time to time to secure solution. Solution must be complete before allowing the evaporation to go too far, or it will be found almost impossible to dissolve the last particles of steel.

The solution is baked as usual in phosphorus determinations, 20 c.c. HCl added and again taken to dryness, taken up in 20 c.c. HCl again, diluted and filtered from tungsten and silicon, which may be ignited and weighed as a check on the first determination. To the hydrochloric acid solution 35 c.c. strong ammonia is added, then sufficient strong nitric to re-dissolve the hydrate of iron: 100 c.c. molybdate solution, made according to Wood's formula as given by Blair, is added to the flask, which is then shaken for a few minutes to ensure complete precipitation of the phosphorus. After standing for an hour the yellow precipitate is filtered on a dried weighed paper, washed with dilute 1 per cent nitric acid, dried for an hour, and weighed as phospho-molybdate, containing 1.63 per cent phosphorus.

For carbon, 1.5 grms. are dissolved in 100 c.c. of a 33 per cent copper and potassium chloride solution. After standing half an hour 5 c.c. HCl is added to hasten solution. When all the precipitated copper has re-dissolved, the solution is filtered through ignited asbestos in a platinum filter tube, using suction to hasten filtration. The carbon on the plug is washed a few times with hot water, then dried and burnt in a platinum tube with a stream of oxygen. The CO_2 is absorbed as $BaCO_3$ in a barium hydrate solution contained in a ten-bulb absorption tube, filtered, washed well with freshly boiled distilled water, ignited, and weighed as $BaCO_3$ containing 6.09 per cent carbon.—*Proceedings of Engineers' Society of Western Pennsylvania*, xvi., No. 4.

ESTIMATION OF ALKALI CARBONATES IN THE PRESENCE OF BICARBONATES.*

By FRANK K. CAMERON.

Introduction.

IN an aqueous solution sodium carbonate is hydrolysed to a definite extent, depending upon the concentration and temperature conditions. This may be represented thus:—



Of the four electrolytes then present in the solution only the sodium hydroxide is dissociated or ionised to any considerable extent, and in consequence the solution presents the characteristic features of a solution of this substance—it is markedly alkaline. Shields (*Ztschr. Phys. Chem.*, 1893, xii., 167), has found the amount of this hydrolysis for a tenth-normal (N/10) solution of sodium carbonate at 25° C. to be about 3.17 per cent. For certain purposes it is desirable to determine the "alkalinity" of such a solution, that is to say, the amount of sodium ions which may possibly result from this hydrolytic action, or one-half the sodium which has been brought into the solution as sodium carbonate. This may be done by titrating the solution with a standard acid solution. But this procedure usually requires that the solution should be heated to the boiling temperature. At the ordinary temperatures the acid reacts with the sodium carbonate to some extent to form the bicarbonate, thus:—



and this formation of the bicarbonate may possibly be augmented by some of the liberated carbonic acid acting on the still undecomposed carbonate. Acid sodium carbonate is itself neutral towards indicators, and in consequence totally misleading results are inevitable. Furthermore, the presence of bicarbonates in the solution, other than that formed by the hydrolytic action of the water, will render an estimation of the sodium alone utterly valueless. The problem has been presented in this laboratory to estimate the amount of sodium carbonate in mixtures containing also the bicarbonate, and, further, to do this without heating the material. Many attempts have been made by others to devise a method for this purpose. That proposed by Winkler has probably proved the most satisfactory. A good description of it has been given by Küster (*Ztschr. Anorg. Chem.*, 1896, xiii., 127). But this method was not adapted to our purposes for several reasons. The method of Sundström described by Lunge (*Ztschr. Angew. Chem.*, 1897, 41), as well as that devised by Lunge (*Ztschr. Angew. Chem.*, 1897, 169) himself, were also found to be impracticable under the conditions which confronted us. Without going into greater detail it may be said that no method of which a description could be found in the literature was free from serious objections. This appeared most surprising in view of the probable technical value of such a method in the manufacture of sodium carbonate. The problem has been satisfactorily solved, and an account of the preliminary work on it may be found elsewhere (*Report No. 64*, U.S. Department of Agriculture, Division of Soils). It was deemed advisable, however, to give the method a more critical examination. The results are recorded in this paper.

Acid potassium sulphate is a well-characterised strong acid. With sodium carbonate it has been shown to react as here indicated:—



The reaction-products, sodium bicarbonate and sodium potassium sulphate, are neutral towards the ordinary indicators. Therefore by titrating a solution containing sodium carbonate with a standard solution of sodium or potassium bisulphate, the amount of sodium carbonate present can be determined directly. Obviously the same statements may be made regarding potassium carbonate. Many indicators have been used with this method, but it may be said at once that, while good results can be obtained with others, phenolphthalein lends itself pre-eminently to the purposes here in view, and it alone is now used in this work in this laboratory. It is to be regretted that the reverse procedure from that just stated cannot be followed, for to the majority of analysts it would certainly be easier to titrate to the appearance of colour rather than to its disappearance. But in this case such a procedure is entirely inadmissible because the sodium carbonate, on being brought in contact with an excess of the strong acid, is more or less decomposed, with the evolution of carbon dioxide, and misleading results that are not comparable are obtained.

It has become evident, in the course of the investigation, that acid sodium carbonate is a very unstable salt, especially in water solutions. The sodium carbonate solutions which had been titrated to loss of colour, immediately began to colour again on standing, the rate of this "inversion" being a function of the concentration and the temperature, as well as time. Some solutions which had been titrated just to loss of colour at 1° C., had practically no colour at the end of an hour, but on being gradually warmed over a Bunsen flame very soon became strongly coloured from the reaction of the regenerated sodium carbonate on the phenolphthalein present. A tenth-normal solution, titrated just to loss of colour, at the room temperature (about 25° C.) will show a marked pink colour within five minutes, and a strong colour within half-an-hour.

A solution of sodium carbonate was divided into a

* Contribution from the Division of Chemistry, U.S. Department of Agriculture. From the *American Chemical Journal*, vol. xxiii., No. 6.

number of portions in small Erlenmeyer flasks, and coloured by the addition of phenolphthalein or litmus. Carbon dioxide was passed in until the solutions no longer showed any alkaline reaction with the indicators. They were then allowed to stand for several days. Some of the flasks were closed with rubber stoppers. The open flasks very soon showed a strong alkaline reaction. In the closed flasks, while a faint alkaline colour appeared within a very short time, the colour became more intense, but very slowly, showing the influence of the carbon dioxide in retarding the inversion. Nevertheless, it would appear that the inversion does take place, even though some carbonic acid must be present. This phase of the subject is now being studied in this laboratory, and the investigation will be continued as time and opportunity may permit.

In a qualitative sense precisely similar results were obtained with potassium carbonate and with sodium silicate, of which both yield acid salts which are unstable in water, and at once invert to a greater or less extent. Sodium borate and disodium phosphate, being salts of weak acids, give an alkaline reaction in water solutions, and can be very conveniently titrated to neutrality with acid potassium sulphate, but in neither case was any subsequent inversion observed.

Description of Experiments.

After some preliminary work it was deemed advisable to test the method by referring all solutions to a standard alkali solution, rather than by making the numerous gravimetric determinations which would otherwise be required. All the titrations were made from two burettes which previous experience had shown to be quite reliable. It was not thought necessary to calibrate them. The burettes were graduated to tenths (0.1 c.c.) and smaller readings could be estimated. It was thought preferable, however, not to attempt readings closer than one-half a scale division (0.05 c.c.), but to depend upon the average of a series of readings.

The standard for reference was a solution of potassium hydrate, accurately prepared and carefully freed from carbonates or other impurities. It was so prepared as to contain 18.17106 grms. of potassium hydroxide per litre. A solution of approximately tenth-normal acid potassium sulphate was then made up and compared with the standard potassium hydrate solution. It was found, as a result of a satisfactory series of titrations, that 1 c.c. of the potassium hydrate solution was equivalent to 6.764 c.c. of the acid potassium sulphate solution. It follows that 1 c.c. of the acid potassium sulphate solution contained 0.006518 gm. of the acid salt, whereas a tenth-normal solution (N/10) would contain 0.006738 gm.

Reasonably pure potassium bisulphate is not difficult to obtain. But one cannot always be certain that an otherwise satisfactory sample contains precisely those proportions of the elements involved, which are required by the formula HKSO_4 . A small excess of either sulphuric acid or the potassium sulphate will not materially alter the value of the reagent for the purposes under discussion, but it is obvious that for very accurate work it is safer to determine the concentration of the solution in the manner just described, rather than depend on either a gravimetric determination of the sulphuric acid alone or of the potassium it contains.

A solution of potassium carbonate (approximately tenth-normal) was then prepared and titrated with the results here given, the first column indicating quantity of potassium carbonate, the second column the quantity of potassium bisulphate, and the third column the ratio of the readings:—

TABLE I.

10.00	12.70	1.270
15.00	18.90	1.260
15.00	18.90	1.260
20.00	25.20	1.260
20.00	25.20	1.260

1.262

These titrations were made in the usual manner by adding a little of the acid solution, shaking, and waiting a few moments to see if colour disappeared before proceeding.

The potassium carbonate was then analysed in the following way:—The solution was treated with an excess of hydrochloric acid, boiled to drive off all the carbon dioxide liberated, and the excess of acid determined by titration with the standard potassium hydrate solution. The figures follow. The first column represents amount of potassium carbonate, the second column hydrochloric acid, and the third column potassium hydrate:—

TABLE II.

40.00	20.00	7.15
40.00	20.00	7.15
40.00	20.00	7.20
		7.166

By a careful and satisfactory series of titrations 1 c.c. of the hydrochloric acid solution was shown to be equivalent to 1.0464 c.c. of the potassium hydroxide solution. Therefore:—

20 c.c. HCl solution	= 20.928 c.c. KOH solution.
Excess " "	= 7.166 " " "
40 c.c. Na_2CO_3 "	= 13.762 " " "
1 " " "	= 0.344 " " "

It has been shown that 1 c.c. KOH solution was equivalent to 6.764 c.c. HKSO_4 solution; therefore, 0.344 c.c. KOH solution was equivalent to 2.327 c.c. HKSO_4 solution. But since only one-half as much acid potassium sulphate is required to convert the potassium carbonate to bicarbonate, it should have required 2.327 + 2, or 1.163 c.c., instead of 1.262 c.c. as actually found. This disagreement was startling in view of the good results previously obtained with the method.

A sodium carbonate solution of about the same strength as the potassium carbonate solution just described was prepared and a long series of titrations made in the manner as with the potassium carbonate solution. It was found that 1 c.c. of the carbonate solution was equivalent to 1.137 c.c. of the acid sulphate solution, though an analysis made in the same manner as in the case of the potassium carbonate showed that 1.035 c.c. of the acid sulphate solution should have been required. The disagreement was practically the same in both cases.

(To be continued).

THE USE OF CARBIDE OF CALCIUM IN METALLURGY.

As is well known, carbide of calcium is principally used for the manufacture of acetylene gas; the steady increase of this means of lighting justifies the existence of the numerous installations which have been erected, especially in the Alps, with a view to the production of large quantities of this substance.

It is, however, a matter of some importance to this new industry that the substance in question should find some other applications, and with this end in view we think it advisable to point out the part it is able to play in metallurgy.

Carbide of calcium is a deoxidising agent; if therefore to a metal in the state of fusion, which is always slightly oxidised, carbide of calcium is added, either alone or with quicklime, the general reaction should be as follows:—
 $\text{CaC}_2 + 2\text{MO} = 2\text{CO} + \text{Ca} + 2\text{M}.$

It is evident that under these conditions, as one part only of the CaC_2 takes part in the reaction, the utilisation of this body would not be very great. To increase its

power, a metallic chloride, RCl , must be added, the chlorine of which will combine with the Ca set at liberty in such a manner that the two affinities of the C for the O , and of the Ca for the Cl , will act simultaneously:— $\text{RCl} + 2\text{MO} + \text{CaC}_2 = \text{R} + 2\text{M} + \text{CaCl} + 2\text{CO}_2$.

These reactions may be applied either to the extraction of a metal from its ores or for the manufacture of metallic alloys, according to whether the metallic chloride taken corresponds with the metal or not.

If $\text{M} = \text{R}$, we obtain the metal M only; if not, we obtain the alloy $\text{R} + 2\text{M}$. We may quote as an example of the last formula the preparation of aluminium bronze, which consists of gently heating a mixture of alumina and chloride of copper, in contact with carbide of calcium.—*Revue Générale des Sciences*, June 30, 1900.

RESEARCHES ON GOLD.

THE ESTIMATION OF GOLD, AND ITS SEPARATION FROM PLATINUM AND IRIIDIUM.

By L. VANINO and L. SEEMANN.

THE method consists in treating the solution containing the metals with peroxide of hydrogen after the addition of an alkaline lye. While other methods require several hours to effect a complete reduction, in this case the gold is precipitated in a few minutes, even in the cold, as a black deposit which under the action of heat agglomerates and becomes of a reddish brown-colour:—



In the case of dilute solutions it is best to apply heat after the precipitation, then acidulate with HCl . For the estimation of gold in commercial chloraurate of sodium it is, however, preferable to effect the reduction by means of formic aldehyde instead of peroxide of hydrogen.

The reaction of peroxide of hydrogen in alkaline solution is much more sensitive qualitatively than any other reaction of gold. With 3 milligrams of gold per litre we can still perceive a pale reddish colouration, appearing blue by reflected light; this would not be detected by other reagents.

Silver is also precipitated quantitatively under the same conditions, but platinum as well as iridium remains in solution; this affords an excellent method for separating these two metals from gold.—*Berichte*, vol. xxxii., p. 1698.

NOTICES OF BOOKS.

Annual Report on the Year 1899. E. MERCK. Darmstadt. March, 1900. Pp. 177.

THE kindly reception which this publication has met with during the last ten years has necessitated a continuous increase in the edition, which has now reached a total of 30,000 copies.

Since the last report the author states he has produced a satisfactory tuberculosis toxin, both from bouillon and from the bacteria (*German patent*, 108,593), thereby nearing the solution of the antituberculosis question. The preparation of this toxin rests upon the fundamental view that both the poisons which pass into solution in the bouillon and those remaining in the bodies of the bacteria should jointly be employed as the initial material for the treatment of tuberculosis: a preparation of this kind has been introduced under the name of "tuberculol."

Another novelty introduced by Mr. Merck is an antidote apparatus for use in cases of poisoning by prussic acid and cyanide of potassium. One method of dealing with cyanic poisoning is by resorting to artificial respiration, evacuation of the stomach, the use of stimulants,

&c.; this, however, has proved successful in light cases only. A second method consists in the use of nitrate of cobalt: the injection of large doses of this salt may cause a diminution, or even disappearance, of the symptoms of cyanic poisoning, although the fact cannot be explained chemically. The third method is based on the conversion of the hydrocyanic acid into a haloid compound. The fourth method involves the principle of the conversion of prussic acid within the system into oxamide by means of peroxide of hydrogen, according to the formula $2\text{CNH} + \text{H}_2\text{O}_2 = \text{C}_2\text{O}_2\text{N}_2\text{H}_4$. This method is the most successful of the four mentioned, and the apparatus devised is for its special application.

There is also a full description of a large number of preparations, some of which have already been noticed in these columns under the heading of "Merck's Digest."

The book concludes with four excellent indices, viz., a general index, a bibliographical index, one of authors' names, and one of diseases, symptoms, and indications for treatment.

The Bacteriology of Every-day Practice. Medical Monograph Series, No. 2. By J. O. SYMES, M.D., D.P.H., &c. London, Paris, and Madrid: Baillière, Tindall, and Cox. 1900. Pp. 90.

THE rapid progress made in modern bacteriology, and its growing importance in medical diagnosis, renders it absolutely necessary that both practitioners and students of medicine should have, at the very least, an elementary knowledge of the methods of bacteriological investigation. In this volume the author's endeavour has been to supply a ready means for the acquisition of bacteriological knowledge, the principal objects in view being to point out in what cases bacteriological examination might help in clinical diagnosis; to describe methods of securing and identifying microscopic specimens such as the student could prepare for himself; and to give directions for taking cultures and for preserving tissues to be sent to the laboratory. We must, however, point out that, assuming as he does that the reader has no elementary knowledge of bacteriological work, the author does not begin sufficiently near the true beginning of the subject; after a few remarks on very simple apparatus, such as a platinum loop (illustrated), a pair of forceps (illustrated), and a glass pipette (illustrated), he plunges straightway into such delicate and advanced work as the staining and mounting of bacteria for microscopical examination. In the succeeding pages he deals with general infections of the blood, suppurative processes, various diseases, and concludes with an appendix on the microscope.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxii., No. 2, July 9, 1900.

Combustible Gases of the Air: Air from the Sea. Existence of Free Hydrogen in the Terrestrial Atmosphere.—Armand Gautier.—From this research and previous researches on the same subject, the author concludes that pure air contains normally about $2/10,000$ of its volume of free hydrogen, to which must be added—owing to the exhalations and fermentations of the soil, vegetables, animals, or human industries—a certain proportion of hydrocarbons—which is relatively great in populous towns, small in country districts, very slight on

rocky plateaux and peaks of high mountains, and practically absent in the higher regions of the atmosphere. There still remain to be determined the nature of the hydrocarbons of the air of towns and woods and the origin of atmospheric hydrogen.

Chemical Constitution of Steel. Influence of Tempering on the State of Combination of Elements other than Carbon.—MM. Carnot and Goutal.—Continuing their researches on the chemical constitution of steel, the authors have now investigated the effect of tempering on modifying the state of combination of the various elements contained in the products of siderurgy. Their new determinations have been carried out with regard to sulphur, phosphorus, arsenic, copper, and nickel.

An Ammoniacal Chromous Sulphate.—Ch. Laurent.—The sulphates of the magnesium series give, with the alkaline sulphates, double salts of the type $MgSO_4 + K_2SO_4 + 6H_2O$. The chromous salt, of analogous formula, known at present, is the double sulphate of chromium protoxide and potassium, $CrSO_4 + K_2SO_4 + 6H_2O$, which was prepared by Peligot. The author has prepared the double ammoniacal sulphate. Owing to the fact that all the chromous salts are very easily transformed into chromic salts when in contact with air, all the experiments had to be conducted in an atmosphere of carbonic anhydride. Analysis shows the new substance to have the formula $CrSO_4 + (NH_4)_2SO_4 + 6H_2O$.

Journal de Pharmacie et de Chimie,
Series 6, Vol. xi., No. 6.

Seleniferous Sulphuric Acid.—MM. Schlagdenhauffen and Pagel.—Already inserted.

The Alteration of Syrup of Iodide of Iron and a Means of Preventing it.—M. Debraye.—The alteration of syrup of iodide of iron is due to the action of the oxygen of the air, which transforms the ferrous salt into ferric salt. To prevent this alteration it has been proposed to add a small quantity of tartaric acid; this, however, only retards the action, but does not prevent it. Formerly, syrup of iodide of iron was kept away from the light; this, however, is quite a mistake. Solutions of ferrous salts should always be put in flasks made of white glass and kept in the light, but they should at the same time be kept full and perfectly stoppered; a small quantity of tartaric acid may be added to aqueous solutions. To show the value of this treatment, the author placed in a 250 c.c. flask 1.35 grms. iodine, 0.66 gm. iron filings, 0.33 gm. tartaric acid, 330.20 grms. simple syrup. The tightly-stoppered flask was placed on a window-sill. In less than forty-eight hours all the iodide was dissolved, and the liquid took the colour of ferric salts. Gradually this colour became fainter, and in fifteen days, in winter, the syrup was as colourless as water, and the whole of the ferric iodide had passed to the ferrous state.

Eugenol, Safrol, and Propylpyrocatechin.—R. Delange.—Already noticed.

Action of Heat on Papain.—V. Harlay.—The digestive power of papain is not affected by dry heat; that is, three hours at 100° over sulphuric acid. The action of heat on papain in solution is very different, however; a temperature of 82°, under the conditions of experiment described, causing its destruction. At temperatures below 75°, the disaggregation of fibrine was complete in twenty-four hours, but at 81.5° the fragments of fibrine were only softened superficially, and at 82° the papain had no effect whatever, even after twenty-four hours.

Russian Pharmacy.—P. Jacob.—Not suitable for abstraction.

No. 7.

This number contains no matter of chemical interest.

No. 8.

Purification of Water by means of the Halogens.—F. Malmjac.—Chlorine has been used both by Traube and Bassenge for the purification of water; the latter recommended a dose of 0.0978 grm. per litre for ten minutes. Bromine, recommended by Schimburg, gave the same result with 0.04 grm. per litre, after five minutes contact. According to Allain, a dose of 1/100,000th part of iodine kills all the non-sporadic pathogenic germs in half-an-hour, as well as most of the saprophytes. The present author has carried out a systematic series of experiments with a badly contaminated water, using varying equal quantities of chlorine, bromine, and iodine per litre, taking the lowest quantity given by the above mentioned authors as the basis; that is to say, 0.01 centigram. per litre of water for half-an-hour. He found that the number of microbes per c.c. was reduced from 17,500 in the original water to 300 by the action of chlorine, to 190 by bromine, and 90 by iodine.

Detection of Benzene in Regenerated Alcohols.—G. Halphen.—For detecting the adulteration of alcohols the author has obtained very good results by forming diazoic compounds, which, conjoined with the naphthols, give oxyazoic colouring matters having so great a colouring power that their detection becomes a matter of extreme sensitiveness. The operation may be divided into five phases:—1. Separation of the hydrocarbides from the alcoholic solution. 2. Nitration of these hydrocarbides. 3. Transformation into amines. 4. Formation of a diazoic salt. 5. Conjunction with a phenol. The author then proceeds to describe the details of the operations. The higher homologues of benzene, under the same conditions, also give colouring matters which indicate their presence.

No. 9.

This number is largely taken up with the death and the funeral orations given at the funeral of the late Gustave Planchon, President of the Committee of the *Journal de Pharmacie et de Chimie*. The death of Prof. Milne-Edwards, professor of zoology at the Ecole de Pharmacie, Paris, is also announced and commented upon.

A Hydrogazometer and Urometer.—E. Benoit.—The author describes two special pieces of apparatus which, however, cannot well be understood without reference to the accompanying diagrams.

Nos. 10 and 11.

These two numbers contain no matter of chemical interest.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xliii., No. 6.

The Electrolysis of Chloride of Potassium.—A. Brochet.—Already noticed in this column.

The Action of Hydrogen on Selenide of Mercury and the Inverse Reaction.—H. Pélabon.—Crystallised selenide of mercury is attacked by hydrogen gas at temperatures above 400°; seleniuretted hydrogen is formed, and at the same time the mercury is set free. To study this reaction the author sealed up selenide of mercury and hydrogen in a tube, and kept them at a constant temperature for some time; they were then cooled quickly, and the gaseous mixture was analysed. After being heated to 440° for several days the gas formed smelt strongly of seleniuretted hydrogen, but the proportion of this gas was found to be never more than 0.6 per cent, no matter how long the heating had lasted. The reaction is thus limited, and this limit is due to the inverse reaction of the seleniuretted hydrogen on the mercury vapour. To show this, it suffices to maintain a sealed tube containing selenide of mercury and hydrogen at a pressure of 760 m.m. for a few minutes at a temperature of about 540°. Under these conditions it is shown that there is 15 per

cent of seleniuretted hydrogen present; but after heating for two days at 440° the proportion of seleniuretted hydrogen is reduced to 0.52 per cent.

The Composition of Essence of Sandal-wood from the East Indies.—M. Guerbet.—Already noticed in this column.

A New Gas-holder for Constant Pressures, Variable at will.—J. Riban.—The advantages claimed for this new gas-holder, which is fully explained and illustrated, are—exact constancy of flow, owing to constant hydrostatic pressure; facility of varying this pressure at will as well as measuring it; facility of completely removing all traces of air or previous gaseous residues; and facility of preventing losses of gas, or entry of air resulting from variations of the external temperature.

MISCELLANEOUS.

Preparation of Ethyldichloramine.—H. Paloma.—Ethyldichloramine has been prepared by several chemists, particularly by Tcherniak, by distilling chlorhydrate of ethylamine with chloride of lime; he has described it as an oil which can be preserved under water, and which distills at $88-89^{\circ}$. The author has also prepared it by this method, but he never succeeded in obtaining a substance which did not decompose; however, by using the dry chloride instead of using a paste of chloride of lime and water, he was successful. The purification of the product thus obtained is much more simple and the return is very good. This ethyldichloramine has been preserved under a thin layer of water in diffused daylight for eighteen months without decomposing.—*Berichte*, xxii., p. 3343.

A New Method for the Estimation of Oxalic Acid in Urine.—E. Salkowski.—This process is based on the property possessed by oxalic acid of dissolving easily and abundantly in ether while phosphoric acid is insoluble in this liquid. Take 200–500 c.c. of urine, add 20 c.c. of HCl (sp. gr. 1.12), and shake this mixture three successive times with alcoholised ether (5 to 10 per cent of alcohol). Decant the layer of ether, filter, distil off nearly the whole of the ether, then after having added a little water, concentrate to 20 c.c. Allow to cool, and separate the resinous residue by filtration, make the solution slightly alkaline with ammonia, and add 1 to 2 c.c. of a 10 per cent solution of chloride of calcium, and acidulate with acetic acid. The precipitate of oxalate of lime thus formed is treated in the ordinary manner.—*Centralbl. f. d. Med. Wiss.*, 1899, No. 16.

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The Qualifying Examination of persons desirous of being registered as PATENT AGENTS will be held in November next.

A Notice giving full particulars has appeared in the "Illustrated Official Journal" (Patents) of July 4th, 1900, and a copy of such Notice can be obtained on application to the Secretary, Chartered Institute of Patent Agents, 19, Southampton Buildings, London, W.C.

PATENTS, DESIGNS, AND TRADE MARKS ACTS 1883 TO 1888.

NOTICE IS HEREBY GIVEN, that DR.

HEINRICH VON NIEDERHAUSEN, of 7, Markkircherstrasse, Rappoltswiller (Alsace), Germany, has applied for leave to amend the Specification of Letters Patent No. 10293 of 1899 for "A Process for Fixing Alumina or Chromic Oxide on Textile Fibres as Mordants, particularly for Dyeing in Adrianople or Turkish Red."

Particulars of the proposed Amendment were set forth in the "Illustrated Official Journal" (Patents) issued on the 1st of August, 1900.

Any person, or persons, may give notice of opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

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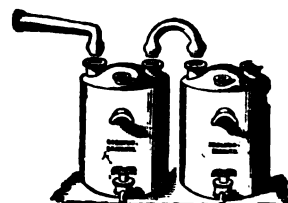
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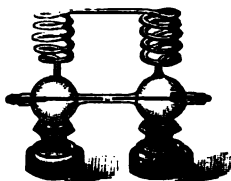
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2125.

THE SEPARATION AND ESTIMATION OF SMALL QUANTITIES OF COBALT IN PRESENCE OF NICKEL.

By THOMAS MOORE, Nouméa.

METALLIC nickel of commerce invariably contains cobalt, never, however, in large quantities, and sometimes only in that delightfully vague quantity termed traces. Up to within a few years ago it was customary in assaying to include the cobalt with the nickel; perhaps this was brought about by the facility of the electrolytic method of analysis, or perhaps by the idea that cobalt formed similar alloys to those of nickel, and might even replace it. This state of affairs, however, exists no longer, and cobalt must now be regarded as an impurity; consequently its accurate estimation becomes a necessity.

Many of the processes for the separation of the two metals, which succeed admirably when the quantity of the two metals is neither too small or disproportionate, utterly fail when applied to minute quantities of cobalt in presence of large amounts of nickel, the precipitations being either incomplete or when complete the precipitates are impure. It was therefore with the object of avoiding some of the difficulties and economising time that the process as given below was worked out.

The volumetric methods for the estimation of cobalt by titration of the higher oxides never seem to have gained confidence, owing probably to their alleged varying composition. Bailey, for example, from a series of experiments, concludes that cobalt solutions precipitated by a hypochlorite give an oxide corresponding to Co_2O_3 ; Carnot, on the other hand, maintains the formula Co_2O_3 ; whilst a whole host of text-books support the popular, but undoubtedly false, composition of Co_2O_3 . Doubtless much depends upon the mode of preparation, but enough has been said to show that there exists a certain doubtfulness as to the exact composition.

Given, however, a solution containing cobalt and nickel, nothing is gained by precipitation with bromine or chlorine and an alkali, since nickel sesquioxide is also thrown down; if, however, an emulsion of oxide of zinc is substituted for the alkali, the cobalt is precipitated almost immediately, the nickel being only thrown down to a slight extent later on and after prolonged boiling, providing always that bromine has been employed. Iodine acts similarly, but more slowly and imperfectly, whilst chlorine causes a complete precipitation of both sesquioxides. When the solution is neutral and contains no zinc salts, the decomposition takes place at ordinary temperatures; in presence of much zinc it is necessary to heat, or even boil, the solution, to obtain a complete separation. By this means it is therefore easy to concentrate the whole of the cobalt mixed with but very little nickel. Having thus obtained such a precipitate, which contains the excess of oxide of zinc, advantage is taken of the dissimilar behaviour of the two sesquioxides to peroxide of hydrogen: this instantly reduces the nickel compound to the green hydroxide, whilst the cobalt compound, whatever may have been its former composition, is now transformed into the true sesquioxide, Co_2O_3 . In actual practice the process is carried out in the following manner:—

The solution containing the chlorides of the two metals should not have much free acid, and if, as is usually the case, a little iron is present, it should be eliminated by precipitation with a slight excess of zinc oxide, and

filtered off. The filtrate is now diluted to from 300 to 400 c.c. with water, one drop of hydrochloric acid added, and raised to the boiling-point or nearly so; now add sufficient bromine water, and then not too much zinc oxide; a large excess only hinders the subsequent filtration, but is not otherwise detrimental. The solution is now boiled for about five minutes, or until the greater part of the bromine is expelled; then filter, and wash precipitate thoroughly with boiling water. The precipitate is now washed as perfectly as possible from the filter into a beaker glass, the trace adhering to the filter-paper being dissolved off by very weak hydrochloric acid containing a very little sulphurous acid; the filtrate, which need not exceed 5 c.c., being received in a capsule, evaporated to dryness, then dissolved in a few drops of water, and added to the main precipitate. The precipitate is now well boiled to thoroughly disintegrate it, and then allowed to become cold. When cold, 5 to 10 c.c. of peroxide of hydrogen, which have been rendered strongly alkaline with caustic soda, are added, the mixture well stirred, and then boiled for two or three minutes to destroy the excess of peroxide, then allowed to cool, after which it is digested for five minutes in a stoppered bottle with potassic iodide and hydrochloric acid, and the iodine thus set free titrated by means of a very weak solution of sodic hyposulphite. $1 \times 0.46511 = \text{Co}$.

The following results were obtained in presence of 2 grms. of metallic nickel perfectly free from cobalt.

Cobalt employed.	Cobalt found.
0.0001 gm.	0.00012 gm.
0.0004 "	0.00041 "
0.0008 "	0.00078 "
0.0010 "	0.00104 "
0.0020 "	0.00201 "
0.0050 "	0.00500 "

THE SPECTRUM OF RADIUM.

By EUG. DEMARCAY.

M^{ME}. CURIE has sent me a specimen of radium chloride which she believes is the purest yet prepared. The spectrum of the dilute hydrochloric solution shows the following lines:—(1). Those of the platinum electrodes. (2). A faint barium spectrum, which is reduced to the three principal lines, 4554.4, 4130.8, and 3892.2. There is also a trace of the line 4525.1. The line 4554.4 is the only well-defined one. (3). The radium lines already given in my previous paper of November, 1899 (*Comptes Rendus*, vol. cxix., p. 716). In spite of the feeble barium spectrum and the strength of the radium spectrum, I did not observe any new line which could be attributed to radium. However, two nebulous bands, already noticed as weak nebulous lines, have considerably increased in strength. The first begins to be sharp towards 4611.9, and attains its maximum about 4627.5. It is almost symmetrical with regard to this maximum, as the band terminates about 4631.0. This and the following band (which is a little stronger), ought to be clearly visible to the eye, as it shows very distinctly in the photograph. The second band has a sharp beginning at 4463.7, and attains its maximum at 4455. The maximum continues to about 4453.4, and then the band decreases in strength very gradually, appearing to terminate about 4390.0.

The strong lines of radium are very powerful and intense in this spectrum. They are among the most intense I have ever seen, especially the lines 3814.7, 4340.8, and 4683.2.

Examination of the various plates shows that the very weak line 4364.2, which was previously considered as belonging to radium, now appears to be due to platinum ($\lambda = 4364.4$). I am also not certain that the line 4600.3 (also very weak) is due to radium, but cannot yet find to

what element it does belong. It is remarkable to note that the character of the radium spectrum, in the same way as the chemical properties of this element, approaches that of the metals of the alkaline earths.

I have not examined the visible, less refrangible portion of this spectrum with the eye, since this examination could only be performed with considerable loss of the precious material. The examination which I have made this winter, of radiferous barium rich in radium, showed very little; I only saw a single line, very ill-defined, which could be attributed to radium. This line has a wave-length approximately equal to 5665. It is only of medium strength, and very inferior to the ray 4826.3 of the same body.

As a result of this examination I think that the specimen of radium chloride in question can be considered as a little purer than former specimens when we consider the extreme sensibility of the spectrum reaction of barium. —*Comptes Rendus*, No. 4, July 23, 1900.

THE RECOVERY OF CHROMIC ACID FROM RESIDUES CONTAINING CHROMIC OXIDE.

By J. REGELSBERGER.

CHEMICAL industries, as well as that of the textile materials, consume large quantities of chromic acid, and it follows that residues containing chromic oxide are very abundant. It is not surprising, therefore, that for a long time attempts have been made to utilise these residues, especially in the manufacture of alizarine.

The bichromates are the most extensively used, especially bichromate of sodium on account of its lower price, from which salts chromic acid is liberated, either by means of sulphuric acid, or, more rarely, by means of hydrochloric acid. For this reason the greater part of the residues of oxide of chromium are met with in the form of sulphuric solutions, forming the compound commonly called chromium soda-alum.

The method used up to the present for the recovery of chromic acid consisted in precipitating hydrate of chromium, by submitting the residuary lyes to a treatment with lime or magnesia, and calcining the precipitate obtained in the presence of oxide or carbonate of calcium and carbonate of sodium. But as this process brought in its train several disadvantages, and was of relatively high cost, it was considered desirable to find a more simple and cheaper method.

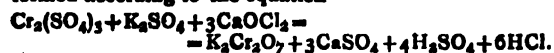
The greatest drawback of this method lay in the loss of the whole of the sulphuric acid of the residuary lyes, so that fresh quantities of sulphuric acid were necessary for the preparation of the bichromates, starting with the products of the fusion, and for their decomposition.

Haussermann (*Dingl. Polyt. Journ.*, No. 283, p. 163) removes a part of this inconvenience by oxidising the alkaline solution of chromite to the state of chromate, by means of electrolysis.

Other methods, due to Lorentz (*Zeit. Anorg. Chem.*, 1896, p. 391) and to Heibling (*French Patent*, 275, 274), utilise chrome-iron as the starting-point.

The methods of Dercum (*English Patent*, 1898, No. 3801) and Fitzgerald (*English Patent*, 1886, No. 5542) are of more interest and appear to have more practical value.

Fitzgerald proposes setting chlorine free by treating the chromic oxide with a mixture of sulphuric and hydrochloric acids, and to recover the chromic acid from the solution of sulphate of chromium thus obtained by means of the electric current. Dercum utilises the well-known reaction which takes place when we treat oxide of chromium with hypochlorite of calcium; chromic acid is formed according to the equation—



Quite recently the firm "Farbwerke-act. Meister, Lucius, and Brünig" (*German Patent*, 103860), have published a process for treating the chromic acid lyes, without losing the sulphuric acid.

The experiments I carried out during the years 1897 and 1898, when the "Farbwerke" patents had not yet been taken out, enabled me to discover the causes to which the trouble brought about by the oxidation of the chromic acid lyes were due. These experiments, which I shall describe in detail, have shown that we may follow two different methods of electrolytic recovery,—that is to say, oxidation in alkaline solution and oxidation in acid solution.

Oxidation in Alkaline Solution.

First of all I examined the oxidising action of hypochlorite of calcium, but the results obtained by the direct action of this substance in the solid state were hardly encouraging. I then tried making the hydrate of chromium to be oxidised into a thick paste, by mixing it either with an alkaline lye, with milk of lime, or with an alkaline sulphate and milk of lime, and submitting the paste thus obtained to the action of a current of chlorine gas. I thus succeeded in oxidising almost the whole of the chromic oxide to chromic acid, but, at the same time, small quantities of hypochlorite or of chlorate were formed, and the residue generally contained a little chromic acid in the form of a calcium salt.

Although the oxidation is quite complete, it does not seem advisable, from the point of view of expense, to have to first prepare chlorine or hypochlorite of calcium. On the contrary, it is desirable to combine in one and the same operation, if possible, the preparation of the oxidising agent and the re-forming of the chromic acid.

Now the electrolysis, without a diaphragm, of solutions of alkaline chlorides is accompanied by the momentary formation of hypochlorites. When consequently, in my opinion, we simultaneously add to an electrolyte of this kind calculated quantities of hydrate of chromium and of salts of chromium, we ought finally to effect the formation of a chromate. This opinion has been fully confirmed.

By the employment of chlorides as electrolytes, we effect, by the sufficiently prolonged action of the current, the integral transformation of the oxide of chromium added into chromic acid. When, on the other hand, we use an alkaline sulphate as the electrolyte, this oxidation hardly takes place, but it becomes manifest immediately on the addition to the electrolyte of a certain quantity of chloride.

With chloride of chromium alone, metallic chromium is formed as well as chlorine. In such a case it is necessary to add a large quantity of chloride to start the oxidation.

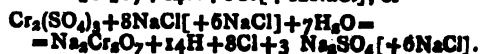
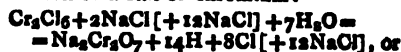
It results from this that the best effect of the current would be obtained when a nearly saturated solution of an alkaline chloride contains a quantity of oxide of chromium equivalent to the current used in the unit of time.

The chromic acid formed is always found in the form of bichromate. When the electrolyte consists of chloride of potassium, the bichromate crystallises in a state of perfect purity from a warm solution, while solutions of bichromate of sodium ought to be previously freed from the chloride of sodium which always accompany them.

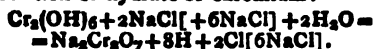
It is not astonishing that this electrolysis is always accompanied by a more or less considerable disengagement of chlorine gas, according to whether we have added to the alkali a salt of chromium or oxide of chromium.

The following equations explain the phenomena in question:—

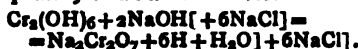
1. Addition of a salt of chromium:



II. Addition of hydrate of chromium:



III. Addition of hydrate of chromium and an equivalent quantity of an alkali or lime.



In this manner the course of the formation of chromate may be represented as follows:—The electrolysis of the solution of chloride causes the formation of hypochlorite, and this immediately oxidises the neighbouring compounds of oxide of chromium, itself changing into chloride. If there is a free base present the chromic acid is neutralised; if not, the chromic acid set at liberty decomposes further quantities of hypochlorite with the evolution of chlorine.

In reality these reactions are not produced so distinctly on account of secondary reactions taking place, such as the reduction of the hypochlorite, its electrolysis, passage to the state of chlorate, &c.

As for the intensity of the current, it should be strongest in the case of a chromium salt, less strong when hydrate of chromium is used (4-7ths of the first), and weakest in the presence of a base in quantity equal to the chromic acid (3-7ths of the first).

On account of the formation of chlorine, the anodes should be formed of some material not attacked by this element. Binoxide of lead and similar substances are not suitable for this purpose, on account of their low conductivity; carbon is strongly attacked, and gives a low return for the current used, so that we are forced to use platinum as being the most suitable.

Although the results obtained by this method are very satisfactory, the oxidation of chromic residues in acid solution offers a more simple means for the recovery of chromic acid. However, oxidation in an alkaline medium is preferable in certain special cases, as, for example, when the chromic lyes contain fairly considerable quantities of organic matter, or, again when the residues are in the solid state.

Oxidation in Acid Solution.

When we electrolyse a solution of sulphate or chloride of chromium, in an apparatus without a diaphragm, we obtain metallic chromium, and, according to circumstances, chlorine gas, but never chromic acid. In the same way, by using a diaphragm and varying the concentration, acidity, and temperature, I found it impossible to get any other result. In all these experiments I made use of platinum electrodes.

A remark made in a treatise on Mineral Chemistry, by Gmelin-Kraut, according to which oxide of chromium can be transformed into chromic acid by means of peroxide of lead in the presence of concentrated sulphuric acid, suggested to me the idea of producing the peroxide by electrolytic means, in the chromic bath itself. For this purpose I made use of lead anodes, and my attempt was crowned with success. I obtained chromic acid by electrolysis of sulphate of lime, and that as easily in the presence of a diaphragm as without one. This electrolysis can be effected either hot or cold, but it is preferable to use a hot solution.

The apparatus necessary is of very simple construction. It suffices to use a vessel of lead, which serves at the same time as anode, and which holds the diaphragm formed of some reducing material, such as lead, iron, nickel, or copper.

The return I obtained from my experiments amounted to 92.5 per cent of theory, with 70 per cent of oxidising action, and to 80 per cent with 86 per cent of oxidising action; these experiments having been made with solutions of pure chrome-alum, with or without sulphuric acid as an electrolyte.

Each electrolysis is accompanied by the formation of a small quantity of white mud, composed of sulphate and

chromate of lead, and at the same time the anode becomes covered with a brownish coating, which, however, exercises no influence on the strength of the current. The power used is, according to my observations, from 8 to 11 kilowatts per twenty-four hours for 100 kilos. of bichromate of sodium. To that must be added the expense necessitated by the construction of the apparatus, and from this point of view it is especially the anodes which must be taken into account.

It is thus probable that with this new method of recovery the cost price of 100 kilos. of bichromate of sodium will not exceed the sum of 20 marks (£1), while 100 kilos. of commercial bichromate costs 54 marks (£2 14s.), without reckoning the sulphuric acid necessary for effecting the oxidation, which acid has always been lost up to the present. — *Zeitschrift für Angewandte Chemie*, p. 1123.

NOTES ON THE HYDROXIDES OF ALUMINIUM.

By E. T. ALLEN.

DURING a study of the best conditions for the precipitation of aluminium by ammonia, I became interested to know the cause of the differences in the density and other properties of the various varieties of aluminium hydroxide. Berzelius, Ramsay, and others have recorded their observations on this subject, but as their statements do not agree, it was thought best to investigate. It was found, in brief, that the composition of the precipitates formed in various ways is the same, viz., $\text{Al}(\text{OH})_3$. The amorphous varieties lose one molecule of water over sulphuric, or at 100° C. The differences, in outward appearance, in density and hygroscopicity are due therefore to differences in physical structure.

I. "Alumina U.S.P." was dissolved in hydrochloric acid, as far as possible, filtered repeatedly until nearly free from turbidity, precipitated in the cold by ammonia, allowed to stand until the precipitate settled, and then thoroughly washed by decantation with cold water.

A. Air-dried (in very hot dry weather).—0.4485 gm. gave 0.1565 gm. H_2O = 34.89 per cent. Calculated for $\text{Al}(\text{OH})_3$, 34.57. (Ramsay found 45.93 per cent; *Journ. Chem. Soc.*, [2], xv., 396).

B. Dried over sulphuric acid.—Prep. I., 0.2594 gm. gave 0.0926 gm. H_2O = 26.31 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09. Prep. II., 0.1323 gm. gave 0.0343 gm. H_2O = 25.92 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09. (Ramsay found 29.5 per cent; *Journ. Chem. Soc.*, [2], xv., 396).

C. Dried at 100° C.—Prep. I., 0.1371 gm. gave 0.0360 gm. H_2O = 26.26 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09. Prep. II., 0.4669 gm. gave 0.1161 gm. H_2O = 24.86 per cent. (Berzelius found 35.5 per cent; "Gmelin-Kraut's Handbuch," 1874—1886, ii., Part 1, p. 619).

II. Aluminium chloride, made as in I., was precipitated by ammonia near the boiling-point, and washed thoroughly by decantation with hot water.

A. Air-dried.—0.2462 gm. gave 0.0833 gm. H_2O = 33.83 per cent. Calculated for $\text{Al}(\text{OH})_3$, 34.57.

B. Dried over sulphuric acid.—Prep. I., 0.2058 gm. gave 0.0545 gm. H_2O = 26.48 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09. Prep. II., 0.2977 gm. gave 0.0771 gm. H_2O = 25.90 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09.

C. Dried at 100° C.—Prep. I., 0.2039 gm. gave 0.0510 gm. H_2O = 25.01 per cent. Calculated for $\text{Al}_2\text{O}_3\text{H}_4$, 26.09. Prep. II., 0.3243 gm. gave 0.0829 gm. H_2O = 25.56.

Ramsay states that the hard horny hydrate precipitated by ammonia contains 45.93 per cent water, and concludes that we have no evidence of the existence of $\text{Al}(\text{OH})_3$.

My experiments showed that the percentage of water depends upon the state of the atmosphere. The preparation I. A., at the end of a hot dry summer, had the composition $\text{Al}(\text{OH})_3$. After several weeks of rainy weather, during which the same preparation was exposed to the air, it was found to contain 36.51 per cent water instead of 34.57 demanded by $\text{Al}(\text{OH})_3$, and a third analysis made about two months later showed that the substance then contained 40.28 per cent water. Another preparation, dried under less favourable conditions than this, gave 39.65 per cent water.

It may be mentioned here that the hydroxide precipitated hot dries to a heavy powder which does not appear to be hygroscopic.

III. The hydrate of aluminium, which is formed by boiling a soluble aluminate with ammonium chloride, is, according to Löwe, much denser and more readily filtered than the precipitate by ammonia, though I have not been able to confirm this statement. Löwe (*Z. B.*, 1860, 132; *Zeit. Chem.*, iii., 247), finds its composition at 100° C. is $\text{Al}_2\text{O}_3\text{H}_4$, like the other varieties of the amorphous hydrate.

IV. W. Crum (*Am. Pharm.*, lxxxix., 156), showed that soluble aluminium hydrate dried at 100° C. contained 25.67 per cent water instead of 26.09 demanded by $\text{Al}_2\text{O}_3\text{H}_4$. Our knowledge of colloidal solutions, however, does not indicate that this is a distinct variety of aluminium hydrate. Pure boiling water will dissolve a considerable quantity of any of the amorphous varieties, at least when freshly precipitated.

V. The hydroxide formed by boiling the basic carbonate with water, or better with water containing a little ammonium salt, is also the trihydrate. Basic carbonate of aluminium was prepared from pure ammonium alum by adding just enough pure caustic potash to re-dissolve the precipitate at first formed, and then saturating the aluminate solution with carbon dioxide. When this preparation was boiled with water it lost carbon dioxide and alkali. Its appearance changed entirely. It became flaky and apparently amorphous, while the basic carbonate was dense and crystalline. The preparation was washed with hot water and air-dried.

0.2393 grm. gave 0.1571 grm. Al_2O_3 .

0.4755 grm. " 0.1642 grm. H_2O .

It was free from alkali.

	Found.	Calculated for $\text{Al}(\text{OH})_3$.
H_2O	34.53	34.57
Al_2O_3	65.05	65.43

This variety is denser and more readily washed and filtered than any of the others except VI.

VI. It has been frequently stated that the compound which carbonic acid throws down from soluble aluminates is the crystalline trihydrate of aluminium. Day (*Amer. Chem. Journ.*, xix., 707), has disproved this, and my experiments confirm his results. The crystalline precipitate formed by the slow action of water on soluble aluminates is, however, crystalline aluminium hydroxide. The following is an analysis of a preparation made by the gradual hydrolysis of potassium aluminate at the boiling temperature:—

0.5194 grm. gave 0.1804 grm. H_2O .

0.1597 grm. " 0.1053 grm. Al_2O_3 .

	Found.	Calculated for $\text{Al}(\text{OH})_3$.
H_2O	34.73	34.57
Al_2O_3	65.93	65.43

The crystalline hydrate of aluminium is a dense crystalline powder which under the microscope was found to consist of minute transparent grains aggregated like strings of pearls. It does not, like the amorphous varieties, lose water over sulphuric acid. A preparation formed by the hydrolysis of sodium aluminate was brought to equilibrium over sulphuric acid.

0.2125 grm. gave 0.0767 grm. H_2O = 36.1 per cent.
Calculated for $\text{Al}(\text{OH})_3$, 34.57.

The dihydrate of aluminium, $\text{Al}_2\text{O}_3\text{H}_4$, is a very hygroscopic powder. It gained water persistently when kept between clamped watch-glasses in a calcium chloride desiccator. One preparation after standing several months in a glass-stoppered weighing glass, which was kept inside a balance-case, had the composition $\text{Al}(\text{OH})_3$.

The conditions under which the monohydrate, $\text{Al}_2\text{O}_3\text{H}_2$, forms have been already investigated by others.

Missouri State School of Mines, Rolla, Mo.

ON ELECTRIC TOUCH AND THE MOLECULAR CHANGES PRODUCED IN MATTER BY ELECTRIC WAVES.*

By JAGADIS CHUNDER BOSE, M.A., D.Sc.,
Professor of Physical Science, Presidency College, Calcutta.

(Concluded from p. 64).

Restoration of Sensitiveness to a Fatigued Substance.

It was said that the inertness of the substance, after long exposure, is due to the presence of a relatively large amount of strained B variety. It therefore follows that if by any means we can transform B into A, then after such a transformation there ought to be a restoration of the sensitiveness. It has also been stated that the B variety under ordinary circumstances is less stable than A. If now we apply a disturbing cause which is unilateral in its action—that is to say, if it converts B into A and not A into B—then such a disturbing cause will re-sensitise the fatigued substance.

Effect of Mechanical Disturbance.—Of the unilateral actions, mechanical vibration is one; for it is known that by the action of friction a substance may pass from one modification to another in one direction only. Thus the change of monoclinic into rhombic variety of sulphur is hastened by scratching with a glass rod, but the change does not take place in the opposite direction. We may now apply the crucial tests. Mechanical vibration will transform B into A, and with positive fatigued substances this ought to produce an increase of resistance (as A is less conducting than B); with negative substances the same disturbance ought to produce a diminution of resistance.

Effect of Heat.—There are other methods by which the B variety may be transformed into A; the more subtle molecular disturbance due to heat may be expected to be even more effective in producing the transformation. Here, too, the crucial test is that by slight heating the fatigued positive substance ought to show an increase of resistance, and the negative substance a diminution of resistance. The two following curves (Figs. 8 and 9) confirm in a remarkable manner my anticipations.

Effect of Heat and Mechanical Disturbances on a Positive Fatigued Substance.—I shall at first deal with the curve for iron. At the end of No. 6 curve, the substance was left in the inert stage b. While in this state, the receiver was heated to a slight extent. Observe in the dotted portion of the curve the sudden fall of conductivity (increase of resistance). I should like to say here, that, though the fall has been indicated by a straight line, as representing the somewhat sudden fall of conductivity, I sometimes noticed on careful inspection a slight oscillatory movement of the galvanometer spot during this process. The significance of this I will notice on a future occasion. The ultimate effect of slight heating (excess of heat produces other complications) is the restoration of the original reduced conductivity. If the application of heat transforms B into A, we may expect the substance to regain its sensitiveness, which it lost in the fatigued state stage b. The receiver was now exposed to radiation, and it at once responded, exhibiting almost its original sensibility. Observe how the subsequent portion of the curve

* A Paper read before the Royal Society, February 8, 1900.

is a repetition of the curve No. 6, and how the substance arrives at the second fatigued state b' . To observe the effect of mechanical disturbance a gentle tap was given to the receiver, and at once there was produced an increase of resistance due to the transformation of B into A, the receiver regaining its sensitiveness by the transformation. The action of radiation was continued, and after a few reversals the substance once more arrived at the third fatigued state b'' . The process described above could be repeated any number of times.

Effect of Heat and Mechanical Disturbance on a Negative Fatigued Substance.

Experiments similar to the above were carried out with an arsenic receiver. From the curve given below (Fig 9) it is seen that the reaction of the negative substance is in every respect opposite to that of a positive substance. It will be noticed that the same cause—i.e., heating or tapping—produces, as necessary consequences of the hypotheses previously stated, two opposite reactions in the two classes of substance. I have been able to verify this deduction by observations with nearly a dozen different substances, and have not, so far, come across any to contradict it. It thus appears that tapping restores the sensitiveness not by the separation of the electrically-welded particles (in which case tapping ought to have

reality. The investigation of this aspect of the subject has given me some extraordinary results; they seem to connect together many phenomena which at first sight do not seem to have anything in common. Another interesting question, the consideration of which has for the present to be postponed, is, why is it that the sensitiveness is so marked in discontinuous metallic particles? In other words, why is the phenomenon mainly one of skin or *twack*? Is the phenomenon wholly unknown in continuous solids?

The experiments described in the present paper show:—

- (1). That ether waves produce molecular changes in matter.
- (2). That the molecular or allotropic changes are attended with changes of electric conductivity, and this explains the action of the so-called coherers.
- (3). That there are two classes of substances, positive and negative, which exhibit opposite variations of conductivity under the action of radiation.
- (4). That the production of a particular allotropic modification depends on the intensity and duration of incident electric radiation.
- (5). That the continuous action of radiation produces oscillatory changes in the molecular structure.
- (6). That these periodic changes are evidenced by the corresponding electric reversals.

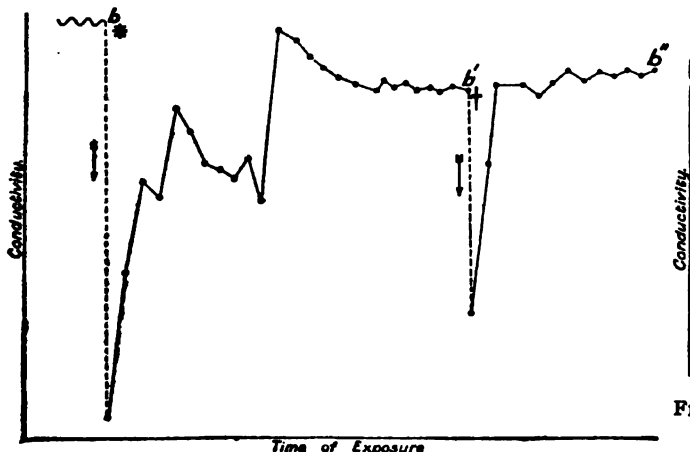


FIG. 8.—CURVE SHOWING THE EFFECT OF HEAT AND MECHANICAL VIBRATION ON A FATIGUED IRON-FILINGS RECEIVER.

* Application of heat.

† Application of a tap.

produced an increase of resistance in both the classes of fatigued substance), but by removing the strain in B and thus converting it into A.

The effect of electric radiation is thus to produce rearrangement of atoms and molecules in a substance; so does light produce new atomic and molecular aggregation in a photographic plate—a subject to be dealt with in detail in a future paper. Some of my audience at the Royal Institution (January, 1897) may remember my attempt at explaining the action of the so-called coherers (which, perhaps, may be better described as “molecular receivers”) by analogy with the photographic action. I had then no proofs for the assertion. I have since been able to obtain experimental evidence that the two phenomena are identical. The coherer may therefore be regarded as a linear photographic plate; since we are more likely to understand the complex photographic action from the consideration of the much simpler action of electric radiation on elementary substances, where the effects are not complicated by secondary reactions, a photographic plate may be regarded as merely an assemblage of “molecular receivers.” I hope also to prove that nearly all the detectors of radiation are molecular receivers in

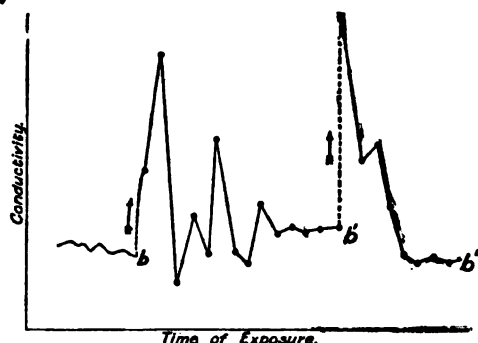


FIG. 9.—CURVE SHOWING THE EFFECT OF HEAT AND MECHANICAL VIBRATION ON A FATIGUED ARSENIC RECEIVER.

* Effect of heat.

† Effect of tapping.

- (7). That the “fatigue” is due to the presence of the “radiation product,” or strained B variety.
- (8). That by means of mechanical disturbance or heat, the strained product can be transformed into the normal form, and the sensitiveness may thereby be restored.

The method described above of detecting molecular changes is extraordinarily delicate, and is full of promise in many lines of inquiry in molecular physics. It is also seen that the phenomenon of contact sensitiveness, contrary to previous suppositions, is perfectly regular. There is no capriciousness in the response of sensitive substances to the external stimuli, which may be mechanical, thermal, or electric. The curves given above show it; but they fail to give a fair idea of the richness and variety of the molecular phenomena, seen as it were reflected in the fluttering galvanometer spot of light; of the transitory variations, of the curious molecular hesitation at critical times as to the choice of the structure to be adopted, and of the molecular inertia by which the newly-formed structure is carried beyond the position of stability, and the subsequent creeping back to the more stable position. The varieties of phenomena are unlimited, for we have in

each substance to take account of the peculiarity of its chemical constitution, the nature of its response to ether waves, the lag and molecular viscosity. All these combined give to each substance its peculiar characteristic curve; it is not unlikely that these curves may give us much information as to the chemical nature and the physical condition of the different substances. I am at present trying to arrange an apparatus which will by means of the pulsating galvanometer spot of light, automatically record the various molecular transformations caused by the action of external forces.

Before concluding, I take this opportunity of expressing my grateful acknowledgments to the Royal Society for the encouragement I received from the Society for the last five years during which investigations on "Electric Radiation" have been in progress at the Presidency College. I may say that the difficulties have been very numerous and disheartening, and that without this encouragement the work which it has been my good fortune to carry out would in all probability have remained unaccomplished. The Government of Bengal has also been pleased to evince a generous interest in these investigations. My assistant, Mr. Jagadindu Ray, and my pupils, Messrs. P. K. Sen, B.A., and B. C. Sen, B.A., have rendered me active assistance.]

THE REFRACTOMETRY OF MINERAL WATERS.

By Dr. E. RIEGLER.

Up to the present time, no special study has been made of the refractive indices of mineral waters. The index of refraction of a solution depends on the nature and the quantity of the salts it contains; this index should therefore be an important character of mineral waters.

Before entering into the details of this work, it is advisable to point out that the index of refraction of a solution varies inversely with the temperature, and, further, that it varies with the adjustment of the refractometer.

To obviate these inconveniences, and to obtain figures independent of the temperature at which the operation is carried out, and of the adjustment of the apparatus, we proceed in the following manner:—

1. We first determine the index of refraction of the mineral water at the temperature shown by the thermometer placed in the cylinder of a Pulfrich refractometer; let N be this index.

2. We then determine the index of distilled water at the same temperature; let n be this index.

The results of this research show that the difference ($N-n$) is independent of the temperature at which the observations are made and of the adjustment of the apparatus. A few examples will demonstrate this fact.

Water from Marienbad (Kreuzbrunnen) at a temperature of 17° has a refractive index $N=1.33500$; distilled water at the same temperature has an index $n=1.33336$. By determining the indices of these same waters at a temperature of 12° , we obtain $N=1.33533$ and $n=1.33369$. The difference of $N-n$ at 17° and at 12° is the same; that is to say, 0.00164.

The most practical refractometer for the determination of the indices of mineral waters is that of Pulfrich; this apparatus will give very exact readings to the fifth place of decimals inclusive.

The temperature is indicated by a small thermometer placed in the glass cylinder containing the mineral water.

The divisions of the refractometer are marked in degrees, which can be transformed into the index of refraction by the aid of a table accompanying the instrument.

The determination is made with a sodium flame, either with a Bunsen burner or Barthel spirit flame.

To determine the relation which exists between the index of refraction and the fixed residue of the mineral water under examination, we evaporated about 100 grms. on a water-bath, then dried the residue at $100-105^{\circ}$ until the weight was constant, and calculated the result for 1 litre.

We can see from the accompanying table that a constant relation exists between the fixed residue of different waters and the index of refraction. It is evident that a knowledge of this index will serve to characterise mineral waters, and thus may be used for their estimation, the more so that these determinations only require a few minutes to perform.

In the accompanying table we have arranged the waters examined according to the amount of their index of refraction.—*Buletinul Societatii de Stiinta d. Bucuresti Romania*, Year ix., Nos. 2-3, p. 251.

ACCIDENTAL IMPURITIES OF CARBIDE OF CALCIUM.

By FELIX B. AHRENS.

IN various samples of carbide from different sources we find large metallic morsels which have been identified as ferro-silicon or silico-carbide of iron. The high proportion of iron in this substance precludes the idea that it is due to impurities in the coke or the lime; it appears rather to be due to the fusion of the metallic clips serving

Table of Mineral Waters Examined from the point of view of their Index of Refraction.

Name of the mineral water.	Temperature.	Index of refraction of mineral waters, N .	Index of refraction of distilled water, n .	Difference between these indices ($N-n$).	Total solid matter in 1000 grms.
1. Eaux-Bonnes	16	1.33369	1.33344	0.00025	0.6621
2. Mont Dore	17	1.33369	1.33336	0.00033	1.3900
3. Caclulata	13	1.33402	1.33361	0.00041	1.8100
4. Ems-Krähchen	17	1.33402	1.33336	0.00066	2.8372
5. Borseck	16	1.33418	1.33344	0.00074	2.9900
6. Wildungen (Helen-Quelle) ..	16	1.33418	1.33344	0.00074	3.3380
7. Niederselters	18	1.33402	1.33328	0.00074	3.3730
8. Biliner-Saurbrunn	18	1.33418	1.33328	0.00090	4.1400
9. Slanic	17	1.33427	1.33336	0.00091	4.9380
10. Karlsbader Sprudel	17	1.33434	1.33336	0.00098	5.4900
11. Karlsbader Mühlbrunn	18	1.33418	1.33328	0.00090	5.5000
12. Vichy-Hôpital	16	1.33451	1.33344	0.00107	5.1750
13. Vichy Grande-Grille	17	1.33443	1.33336	0.00107	5.0530
14. Kissingen-Rakocsi	18	1.33484	1.33328	0.00156	8.7290
15. Marienbad-Kreuzbrunnen ..	17	1.33500	1.33336	0.00164	8.9600
16. Hunyadi Janos	18	1.34003	1.33328	0.00675	43.1100
17. Breas	19	1.34173	1.33320	0.00853	55.8870
18. Carabana	18	1.34475	1.33328	0.01147	75.3270
19. Rabinat	17	1.34692	1.33336	0.01356	100.8700

as holders for the electrode, and this fact seems to indicate a considerable consumption of the electrodes. This inconvenience is, as a rule, due to the faulty construction of the furnace, inasmuch as it allows too great a consumption of the electrodes, or else does not allow of good regulation. It is extremely improbable that this ferro-silicon is due to a mistake in the manufacture, as it is very frequently found in commercial carbide of calcium.

A short time ago I found in a carbide of calcium some of these metallic morsels, a few of which were several c.m. in diameter, and I was able to collect a certain quantity. They contained not only iron and silicon, but also a large proportion of copper. It may easily be imagined that this latter metal came from the conducting-wires and fell into the furnace after a short circuit. This silicide had a crystalline fracture, was fairly friable, and strongly resistant to chemical reagents. Boiling hydrochloric acid had absolutely no action on it, and boiling aqua regia very little. Of a piece weighing 0.5 gm. only 0.1382 gm. was dissolved after boiling three days with this latter reagent. The alkalis and alkaline carbonates attack it very easily when fused.

To effect its analysis we begin by breaking up a few pieces taken at random, in a steel mortar, and then pulverise them in an agate mortar; the powder is then fused with the double carbonate of potash and soda; during this operation a mass of green flames is given off.

We can never manage the complete disaggregation of the silicide in one operation, even after maintaining the fusion for several hours; it is necessary to submit the silica separated by means of hydrochloric acid to the same treatment once or twice more. After completing the operation in the ordinary manner, by making the silica insoluble by means of hydrochloric acid in the presence of a small quantity of chlorate of potash, we precipitate the iron by a large excess of ammonia, so that the copper will remain in solution. The oxide of iron is re-dissolved and re-precipitated three times under the same conditions to get it absolutely free from copper. Finally the copper itself is precipitated by means of sulphuretted hydrogen in hydrochloric solution.

As the silicide contains carbon, this latter is estimated by combustion in a current of oxygen.

0.1595 gm. of silicide gave—

0.006 gm. CO_2 = 1.03 p.c. C.

0.4016 gm. of silicide gave—

0.2362 gm. SiO_2 = 27.61 " Si
0.3384 " Fe_2O_3 = 58.96 " Fe
0.654 " Cu_2S = 12.76 " Cu

100.36 p.c.

We found certain differences on examining the physical properties of various morsels of silicide; some were of a grey colour, and others, more numerous, were slightly red. The first had a hardness of 8 to 9, and were friable and easily powdered; the second had a hardness of 7 to 8, and were more coherent. Finally we met with a small quantity of flattened cakes, grey in the interior and bright yellow on the surface.

These three different substances were analysed by the method described above, and found to have different compositions. The grey portions with the hardness 8 to 9 contained—

0.2642 gm. of the material gave—

0.1884 SiO_2 = 27.00 per cent Si
0.2288 Fe_2O_3 = 60.62 " Fe
0.0154 Cu_2S = 4.66 " Cu
0.0186 $\text{P}_2\text{Mg}_3\text{O}_7$ = 1.97 " P

The remainder consisted principally of carbon.

The analysis of the reddish pieces gave the following results:—

0.6682 gm. of the material gave—

0.3436 SiO_2 = 24.00 per cent Si
0.4924 Fe_2O_3 = 51.58 " Fe
0.1988 Cu_2S = 23.76 " Cu
Remainder = 0.66 " C

As for the flattened cakes, they were analysed after being powdered and thoroughly mixed, and gave the following results:—

0.3475 gm. of the material gave—

0.1453 SiO_2 = 19.6 per cent Si
0.2904 Fe_2O_3 = 58.5 " Fe
0.0956 Cu_2S = 21.9 " Cu
100.0 "

—*Zeit. für Angewandte Chemie*, May 1, 1900.

ANALYSIS OF THE MINERAL WATER FROM THE COLD OR "PARC" SPRING AT EVAUX-LES-BAINS (CREUSE).

By EDMOND BONJEAN.

The mineral water springs at Evaux-les-Bains are as a rule thermal; their temperature is 56° (César), 54° (Desglades), 52° (Bain de Vapeur), and 39° (Bassin Rond). These temperatures were taken by M. Picaut in February, 1900. The temperature of the air at the time was 12°.

Recent excavations have shown the existence of this new spring. Like most mineral springs from a common source, the different springs at Evaux come from the same thermal fissure, form several branches during their passage through the ground, and give rise to several outfalls of different temperatures; this is caused by the cooling undergone by each stream of water during its circulation through the more or less elongated subterranean passages connecting the principal thermal fissure with the surface of the ground.

There is a strong resemblance between the results of the analysis I have made of the water from the cold spring and the results of the analyses made in 1877, at the labora-

Results of the Analyses.—Samples taken December 28, 1899.

The results are given in grms. per litre.

I. Elementary Analysis.

Silica, SiO_2	0.0990
Oxide of iron	traces
Oxide of manganese	traces
Alumina, Al_2O_3	traces
Lime, CaO	0.0728
Magnesia, MgO	0.0090
Soda, Na_2O	0.5498
Potash, K_2O	0.0339
Lithia, Li_2O	0.0020
Ammonia	traces
Sulphuric acid, SO_3	0.5001
Chlorine, Cl	0.1214
Total carbonic acid	0.2402
Arsenic	0.00025
Fluorides	distinct traces
Nitrates, nitrites, iodine	none
Oxygen absorbed from per- manganate of potash .. } Acid sol. ..	0.0035
Residue at 110—120°	0.0035
Residue after incineration	1.492
Loss at red heat	1.421
Sulphuric residue	0.071
Total hydrometric degree	1.550
Permanent hydrometric degree	21.5°
Alkalinity, as CO_3Na_2	2.0°
	0.2968

tory of the School of Mines, of the various thermal waters of Evaux. I have detected rare substances which were not noticed in these former analyses, amongst them being lithium, arsenic, manganese, and fluorine, which are of great therapeutic interest.

The fluorine was detected in 10 grms. of residue (from about 6.5 litres) attacked by pure sulphuric acid, and the products of distillation collected in distilled water. The presence of fluoride of silicon was shown by the production of gelatinous silica on contact with water.

The residue from the water is fairly rich in silica (99 m. grms. per litre), so that it is unnecessary to add silica before treating with sulphuric acid.

The alkalis (soda, potash, lithia) were separated and estimated by the method I have already described (*Bull. Soc. Chim.*, Series 3, vol. xxi., p. 691). The lithia was weighed in the state of phosphate, Li_3PO_4 . The arsenic was estimated in Marsh's apparatus on 10 grms. of the residue.

The iodine, tested for by Armand Gautier's method (*Bull. Soc. Chim.*, Series 3, vol. xxi., p. 456), gave only negative results on 10 grms. of residue.

It should be noted that this water does not contain any free carbonic acid, the whole being saturated with the alkaline earths, the soda and lithia, in the state of bicarbonates.

II. Arrangement of Fixed Elements.

Free silica, SiO_2	0.0990
Carbonate of lime, CO_3Ca	0.1300
Carbonate of magnesia, CO_3Mg	0.0189
Carbonate of soda, CO_3Na_2	0.1350
Sulphate of soda, SO_4Na_2	0.8363
Sulphate of potash, SO_4K_2	0.0627
Chloride of sodium, NaCl	0.2001
Carbonate of lithium, Li_2CO_3	0.0048
Arseniate of soda, AsO_4Na_3	0.0007
Fluorine, alumina, ferrous and manganous carbonates	traces
Total fixed elements	1.4875
Residue at $110-120^\circ$	1.4920

III. Probable Combination of the Elements Found in the Water.

Free carbonic acid	none
Sulphate of soda, SO_4Na_2	0.8363
Sulphate of potash, SO_4K_2	0.0627
Chloride of sodium, NaCl	0.2001
Bicarbonate of soda, NaHCO_3	0.2138
" lime, CaCO_3CO_2	0.1872
" magnesia, MgCO_3CO_2	0.0287
" lithia, LiHCO_3	0.0076
" iron	traces
" manganese	traces
Free silica, SiO_2	0.0990
Alumina, Al_2O_3	traces
Arseniate of soda, AsO_4Na_3	0.0007
Fluorides	distinct traces
Organic matter (as oxygen)	0.0035
Ammoniacal salts	traces
Organic nitrogen	traces
Total matter in solution	1.6396

—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 10.

Action of Reduced Nickel on Acetylene.—Paul Sabatier and J. B. Senderens.—The authors continue their research on the hydrogenation of acetylene in presence of nickel, cobalt, reduced iron, or platinum black, and now prove that, in contact with a column of these metals, maintained at a suitable temperature, the hydrogen produced by the destructive reaction acts on the excess of acetylene.—*Comptes Rendus*, cxxxi., No. 3.

ESTIMATION OF ALKALI CARBONATES IN THE PRESENCE OF BICARBONATES.*

By FRANK K. CAMERON.

(Concluded from p. 69).

Two series of titrations were then made with the potassium carbonate solution. In the first series, the potassium carbonate solution was heated to boiling in each case before titrating. In the second series, in each case the solution was filled with crushed ice and shaken until the temperature was lowered to less than 10°C . before titrating. The number of c.c. of the acid sulphate solution required to neutralise 1 c.c. of the potassium carbonate solution was—

At 0° to 1°C	1.455 c.c.
At room temperature (about 26°C .)	1.262 "
After boiling (about 97°C .)	1.210 "

From these results it would appear that the reaction was more complete at the higher temperature, in spite of the fact that the inversion of the acid potassium carbonate is more rapid at these higher temperatures and might be expected to produce exactly opposite results. For instance, the solutions which had been titrated at 97° were very strongly coloured within five minutes after the titration was completed, while those which were titrated at 10° showed only a faint pink colour after standing for upwards of an hour. The true explanation of the results, however, became apparent in the course of these titrations. It was found that it takes a measurable time for the reaction between the acid sulphate and carbonate to run to end, and that if the acid sulphate is delivered too rapidly from the burette a considerable excess may be run into the carbonate solution before the colour of the indicator disappears, so that, with these two effects of inversion of the acid carbonate and the relatively slow reaction velocity between the carbonate and acid working against each other, it would be possible to run in the solution at such a rate as to obtain any desired result within quite wide limits, and, in fact, beautifully comparable results were thus obtained. The value of the method would be very slight if the personal equation could not be eliminated in the titrations. That this can be done, however, was clearly demonstrated. If the acid potassium sulphate solution is delivered from the burette at about the rate of two drops per second, and the vessel containing the alkaline carbonate is constantly and vigorously shaken, markedly lower reading will be obtained than by any other procedure; furthermore, the readings thus obtained were found to be quite independent of the temperature at which the titrations were made. These facts were confirmed by several long and satisfactory series of titrations. This point having been clearly established, a solution of sodium carbonate was carefully prepared and boiled for some time to complete the inversion of any acid sodium carbonate which might be present. After being cooled to room temperature and made up to the desired volume, it was titrated with the following results:—The first column represents the amount of carbonate, the second column the amount of acid sulphate, and the third column the ratio of the readings:—

TABLE III.

20.00	21.50	1.075
20.00	21.60	1.080
30.00	32.30	1.076
30.00	32.30	1.076

1.077

The sodium carbonate solution was then analysed by boiling with an excess of acid potassium sulphate and titrating the excess of acid with a solution of potassium

* Contribution from the Division of Chemistry, U. S. Department of Agriculture. From the *American Chemical Journal*, vol. xxii., No. 6.

hydrate of known concentration. The results are here given:—The first column indicating amounts of carbonate taken, the second column amounts of acid potassium sulphate, and the third amounts of potassium hydrate required to neutralise the excess of acid:—

TABLE IV.

20'00	50'00	5'50
20'00	50'00	5'50
20'00	50'00	5'50
20'00	50'00	5'50
		5'50

1'00 c.c. KOH solution = 1'352 c.c. HKSO₄ solution.
5'50 " " " = 7'436 " "
20'00 " Na₂CO₃ " = 43'564 " "
1'00 " " " = 2'128 " "

But since only one-half as much acid potassium sulphate would be required to convert the carbonate to acid carbonate, 1 c.c. of the Na₂CO₃ was equivalent to 2'128 c.c. ÷ 2 or 1'064 c.c. of the HKSO₄ solution. Comparing the value found by direct titration, 1'077 c.c., the error for 1 c.c. was about 1'2 per cent. More accurate results have, however, been obtained for both sodium carbonate and potassium carbonate. This error would amount to 0'10 c.c. in reading for 10 c.c., about 0'25 c.c. for 20 c.c., or nearly 0'50 c.c. in reading a titration of 30 c.c. But it has been shown repeatedly that readings for this amount could be obtained by different observers agreeing to within less than 0'20 c.c., and it may be said that the probable error for such an amount is certainly no greater than this. Considering the number and nature of the operations involved, the agreement obtained above was considered satisfactory, and it was not deemed worth while to repeat the work merely for the purpose of being able to present more refined figures.

In order to demonstrate that the presence of sodium bicarbonate in the salt analysed does not affect the accuracy of the method, mixtures of the carbonate and bicarbonate were prepared. Before titrating these mixtures with the acid potassium sulphate solution, the solutions of the carbonate and bicarbonate were separately titrated with this reagent. In Table V. the first column represents amounts of sodium carbonate taken, the second column the amounts of acid potassium sulphate required to neutralise them respectively, and the third column the ratio of the readings:—

TABLE V.

10'00	8'90	0'890
20'00	17'50	0'875
30'00	26'30	0'876
15'00	13'29	0'886
10'00	8'90	0'890
20'00	17'60	0'880
20'00	17'60	0'880
30'00	26'45	0'882
30'00	26'45	0'882
30'00	26'45	0'882
		0'882

A solution of sodium bicarbonate was then prepared and allowed to stand until equilibrium had been reached with the inverted normal carbonate. It was then titrated with the results here given:—

TABLE VI.

25'00	14'60	0'586
10'00	5'90	0'590
10'00	6'10	0'610
10'00	6'10	0'610
20'00	12'30	0'615
		0'602

Mixtures were then made by adding 10 c.c. of the sodium bicarbonate solution to 20 c.c. of the normal

sodium carbonate solution and titrating as before. The first column represents the amount of acid potassium sulphate solution required, the second column gives the reading corrected for the sodium bicarbonate added, and the third column the corresponding amount of acid required to neutralise 1 c.c. of the sodium carbonate solution taken:—

TABLE VII.

23'70	17'68	0'884
23'75	17'73	0'886
23'70	17'68	0'884
		0'885

The agreement of this figure, 0'885, with that found in Table V., 0'882, is very satisfactory, and may be regarded as establishing the point under investigation. It should be remembered, however, that when sodium carbonate is added to a solution containing sodium bicarbonate—and consequently some inverted carbonate—the equilibrium between the two substances may well be materially altered. In solutions as dilute as those examined, this displacement was probably very small, and so did not interfere with the demonstration of the fact that the presence of acid sodium carbonate does not interfere with the estimation of the hydrolysed sodium in the solution. But when the concentrations are considerable, this equilibrium displacement may well become an important factor. Should this method ever commend itself to use in technical work, this displacement of the equilibrium corresponding to an apparent increase of the amount of normal carbonate present on dissolving mixtures must be considered.

An interesting extension of the method has been developed in the course of our work. It is frequently necessary to make a rapid determination of the chloride as well as the carbonates in solution. This may be done in the following way:—As soon as the solution containing the carbonate has been titrated to neutral action with acid potassium sulphate, a drop or two of this reagent is added in excess to retard the inversion of the bicarbonate to the normal alkaline carbonate. A small amount of a solution of potassium or ammonium chromate is then added as an indicator, and the solution titrated at once with a standard solution of silver nitrate. Before titrating with the silver nitrate the solution may be boiled, in which case the inverted carbonate must again be neutralised before making the determination for the chloride. But little advantage is gained thereby, however, and results in every way satisfactory have been repeatedly obtained, working throughout at the room temperature. For instance, a solution (tenth normal) of sodium carbonate was prepared by standardising against a tenth normal (N/10) solution of acid potassium sulphate; also a solution of sodium chloride, 1 c.c. of which was equivalent to 1'734 c.c. of a tenth normal solution of silver nitrate. The following are the results obtained with the mixtures of the sodium carbonate and sodium chloride solutions. The first column represents amounts of sodium carbonate taken, the second column represents the amounts of sodium chloride taken, the third column the amounts of acid potassium sulphate required to neutralise the mixtures, and the fourth column the amounts of silver nitrate required to precipitate the chloride present:—

TABLE VIII.

5'00	10'00	5'05	17'35
10'00	10'00	10'10	17'35
15'00	10'00	15'20	17'35

The agreement shown in these results leaves nothing to be desired, and many more equally satisfactory determinations have been made. An interesting theoretical point is involved in the operation just described. The presence of normal carbonates in the solution would, it is well known, interfere with a titration for chloride with silver nitrate, as insoluble silver carbonate would be

formed to some extent and interfere with the desired precipitation of the chloride. The reaction is to be regarded

as the result of the silver ions Ag^+ coming in contact with the carbonic acid ions CO_3^{2-} . But no such reaction is to be observed in the case we have been discussing, where acid sodium carbonate is in the solution. Therefore it appears reasonable to assume that acid sodium carbonate does not yield a CO_3 ion, but probably dissociates, thus—



and the ion HCO_3^- does not react with the silver ion to give an insoluble compound. There is further evidence to support this view. (Walker and Cormack, *Journ. Chem. Soc.*, 1900, xlvii., 5; "Foundations of Analytical Chemistry," Ostwald and McGowan, pp. 193 and 207).

If a solution of acid sodium carbonate is added to a solution of a barium salt there is only a little precipitate formed at first, though the precipitation of the barium generally proceeds and is completed in time; more quickly if the solution be heated. It has been shown that acid sodium carbonate in water solution is unstable, some normal carbonate being formed at once, and it is to this small amount of normal carbonate that the first precipitation of the barium is due. But when this has taken place the equilibrium between the sodium carbonate and acid sodium carbonate is destroyed, more sodium carbonate is formed, and the precipitation of the barium again proceeds. This action is, of course, continuous. As the inversion of the acid sodium carbonate is more rapid at high temperatures, the formation of the normal carbonate and subsequent precipitation of the barium will proceed more rapidly on heating.

The use of ammonium carbonate in precipitating insoluble carbonates seems worthy of consideration in this connection. Unlike the corresponding sodium and potassium salts, ammonium carbonate is unstable in water solution, breaking down with the formation of the acid carbonate and the escape of some of the ammonia as such. But an equilibrium is established between the normal carbonate and the acid carbonate, which is destroyed when the solution is brought into contact with some salt which will precipitate an insoluble carbonate, such as calcium or barium. The inversion of the acid carbonate is measurably slow, however, and, as is well known, to obtain complete precipitation the solution must be allowed to stand for some time or be heated.

A statement of some of the preliminary experiments on the inversion phenomena referred to may be of interest in this connection. Three portions of a sodium carbonate solution were titrated with acid potassium sulphate to loss of colour with phenolphthalein as indicator, allowed to stand for twenty-four hours, and then titrated a second time to loss of colour. The results are here given, the first column being amounts of the carbonate solution, the second column amounts of acid sulphate added, and the third column amounts of acid sulphate required to neutralise the solution after standing.

TABLE IX.

10'00	8'90	1'00
20'00	17'50	1'85
30'00	26'30	2'55

It would appear that the inversion was approximately proportional to the initial amount of the bicarbonate, but as the concentrations were not quite the same and the sulphates present may have an influence, this conclusion can only be drawn tentatively. Some measurements, which have been made by Mr. Lyman J. Briggs, indicate that the inversion at first approaches a maximum quite rapidly, but, when equilibrium has been nearly reached, it becomes very slow and probably requires a long time before reaching final equilibrium.

Three portions of 20 c.c. of a potassium carbonate solution were each titrated to disappearance of alkaline reaction with 24'2 c.c. of the acid potassium sulphate solution, and, after standing forty-eight hours, each required 3'1 c.c. of the acid sulphate solution to neutralise them.

Ten c.c. of a sodium silicate solution required 14'1 c.c. of the acid sulphate solution to neutralise it. It was immediately boiled for three minutes, after which it required 1'1 c.c. of the acid sulphate solution to neutralise it; another portion of 10 c.c., to which 14'1 c.c. of the acid sulphate had been added after the expiration of an hour, at the room temperature, required 0'9 c.c. to neutralise it. The case of the sodium bisilicate differs essentially, however, from that of the sodium bicarbonate, in that no volatile component can be formed. So that this apparent inversion must be more limited in amount, and is in reality a measure of the hydrolysis of the salt. It was not appreciable in the case of the borates or phosphates, as has already been noted.

An application of the method to a solution of sodium silicate was made. Table X. gives the results of the titrations of the solution with the acid potassium sulphate, the first column indicating amounts of the silicate solution, the second column amounts of the acid sulphate, and the third column the corresponding ratios:—

TABLE X.

20'00	31'00	1'550
20'00	31'00	1'550
10'00	15'70	1'570
10'00	15'80	1'580
10'00	15'50	1'550
10'00	15'70	1'570
15'00	23'60	1'573
15'00	23'60	1'573
20'00	31'40	1'570
20'00	31'45	1'572
		1'566

The solution was then analysed by adding an excess of hydrochloric acid, boiling, and titrating the excess of the acid with a solution of potassium hydroxide. The first column of Table XI. indicates the amounts of silicates taken, the second column the amounts of hydrochloric acid added, and the third column the amounts of potassium hydrate required to neutralise the excess of acid:—

TABLE XI.

40'00	20'00	11'70
40'00	20'00	11'70
40'00	20'00	11'70
		11'70

Since in this case the silicic acid does not escape from the solution as does carbonic acid, when the hydrochloric acid is added to excess, enough potassium hydrate will be required not only to neutralise the excess of hydrochloric acid, but also to re-convert the silicic acid to potassium bisilicate before the solution will be alkaline.

20'00 c.c. HCl solution	=	20'928 c.c. KOH solution.
Excess of "	=	11'700 " "
40'00 c.c. sodium silicate		" "
solution	=	9'228 " "
1'00 c.c. sodium silicate		" "
solution	=	0'231 " "

It has been shown that 1 c.c. KOH solution was equivalent to 6'764 c.c. H_2SO_4 solution; therefore 0'231 c.c. KOH solution was equivalent to 1'562 c.c. H_2SO_4 solution. The agreement of this figure 1'562 c.c. with that given in Table X., 1'566 c.c., must be regarded as entirely satisfactory.

Summary.

The principal results of this investigation may be summarised as follows:—

1. The amount of a soluble alkaline carbonate in a solution can be quickly and accurately determined whether the bicarbonates are present or not.

2. The method seems well adapted to the estimation of silicates, borates, phosphates, and the salts of weak acids in general.

3. The bicarbonates are unstable in water solution, and are more or less completely converted into the normal salt.

4. Alkaline bicarbonates are themselves neutral in water solutions; they do not yield a CO_2 ion by hydrolysis, or they do so only to a slight extent.

5. Therefore, an accurate volumetric determination of chlorides by means of a standard silver nitrate solution is feasible in the presence of alkaline bicarbonates, if the hydrolysis of these latter is prevented.

NOTICES OF BOOKS.

Physics: Experimental and Theoretical. In Two Vols. By R. H. JUDE, D.Sc., M.A., and H. GOSSIN. London: Chapman and Hall. Vol. I. Pp. 926.

THIS work is based upon a book by Prof. Gossin, of Lyons, and has been translated and considerably amplified by Dr. Jude, who is responsible for its present form.

The first volume is divided into three sections, and treats of Mechanics, Hydrostatics, Pneumatics, Heat, and Acoustics, and is intended for use in the senior classes of science and technical schools. The general plan of the work is the same as that followed in Ganot's and Deechanel's "Physics," though a great deal of historical matter has been superseded in favour of more modern material.

It is a matter for regret that Dr. Jude did not devote more time to correcting and revising his proofs, for he has committed himself to several statements which, to say the least, are open to question, especially in the earlier part of the book. For instance, on page 11 we read: "The external force causing the body to travel in a curve must always be in the direction of the normal to the curve (drawn inwards) through the body." This statement is of course incorrect, and is probably intended to refer to motion in a circle. Again, on pages 4 and 5: "It is well known to architects that the lower stones of buildings become compressed by the weight which they support; this is called *settling*." "Settling," as Dr. Jude should know, is due to the displacement, by "compression," or otherwise, of the ground beneath the buildings. On page 26 we are informed that: "On some railways, too, where the rolling-stock is exceptionally good, billiard saloons have been fitted up, and it is possible to play as the train goes along just as if the billiard-table were at rest." It is unfortunate that we are not informed on which railways these saloons are to be found, nor are we favoured with the opinions of the players as to the way the tables "play."

On page 115: "We have, then, the following theorem: *The vertical pressure of a liquid on any immersed surface, of whatever shape, is equal to the weight of the actual column of liquid lying directly above it.*" And this follows a proof on page 113 to the contrary!

Instances of loose and inaccurate statements could be multiplied, but enough have been quoted to show that the sections on "Mechanics" and "Hydrostatics," at any rate, require to be very carefully edited before the next edition is published. The next section, "Pneumatics," contains a good description of the formation of cyclonic and anti-cyclonic areas, and should enable anyone to understand the daily weather-charts published by the Meteorological Office, and to forecast, with a fair measure of success, the probable "weather" in their own locality for twenty-four hours ahead.

The explanation of the supporting action of the aeroplane, on page 176, is not very convincing; Dr. Jude

ignores the influence of the upward slant of the plane altogether; and his reference to a train crossing a rotten bridge in one of Bret Harte's tales is not very apt; for the train could not cross a continuous succession of rotten bridges in the manner indicated.

It is the next section on "Heat," however, on which the book should really be judged, extending as it does over more than 60 per cent of the text; and here it is apparent that Dr. Jude is in his element; for, although there are many indications of hurried writing, we meet with no more of those statements which mar the earlier sections of the book.

Dr. Jude explains the phenomena of heat as manifestations of the internal kinetic energy of the molecules whereof matter is composed, and thence shows the intimate relation between heat and work. In our opinion, he goes too far in this direction, as he constantly refers to so many foot-pounds of heat. Many workers have determined experimentally the ratio between heat units and external work (known as Joule's equivalent); the latest determination by Rowland is (on the British scale), $J=780$. The greater part of this section is devoted to the behaviour of gases under varying conditions of pressure and temperature, whereby the difference between adiabatic and isothermal expansion is explained, and by a natural sequence the theory of entropy is developed. This is from its nature a very difficult subject, and is, moreover, one which is very little known. It is, as Dr. Jude remarks, almost impossible to define it, and it is not, like mass, or time, self-evident as to existence, and only needing precise standards of measurement to make it scientifically definite; but he has succeeded in describing it, in showing how to measure it, and indicated the use of it. On page 619 is described an interesting experiment, whereby a substance can be caused to pass from the state of an ordinary gas to that of an ordinary liquid, or vice versa, without the transition being perceptible at any stage of the process.

The section ends with a history of the kinetic theory of gases; the various modifications adopted from time to time are mentioned, and the numerical results arrived at with regard to the probable mean size and velocity of molecules. No treatment of this branch of physics can be satisfactory without an extensive use of the calculus and higher mathematics generally. Dr. Jude has, however, done very well under his self-imposed limitation.

The final section on acoustics calls for no remarks; it covers the usual ground in the usual manner.

There are numerous examples interspersed through the book, illustrating every theory treated of.

The figures number nearly 400, most of which, except Fig. 3, which ought never to have been admitted, are clear and well-chosen.

There is no index, but one is scarcely needed by reason of the exceptionally full table of contents.

When the blemishes mentioned above are removed, we think there will be a good future before the book.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 3, July 16, 1900.

Radio-activity of Uranium.—Henri Becquerel.—The author employed M. Debièvre's method to try and extract the radio-active material from uranium. This method consists in mixing uranium chloride with barium chloride, and then precipitating the barium as sulphate. This latter salt carries down with it a very active product, whilst the uranium salt remaining in solution is less

active than it was before this operation. If this operation is repeated a great number of times, the active property of the uranium appears to be gradually extracted, but after about the eighth operation the activity of the uranium diminishes very little. Taking the conductivity communicated to air by the original material as unity, the uranium salt after twelve operations is about half, whilst the activity after eighteen operations is reduced again about one-sixth.

Preparation and Properties of two Borides of Silicon, SiB_3 and SiB_5 .—Henri Moissan and Alfred Stock.—Boron and silicon combine directly at a high temperature, producing two crystalline borides of formulae SiB_3 and SiB_5 . These two new compounds are soluble in melted silicon, from which they may be separated by treatment with hydrofluoric and nitric acids. The crystals easily scratch ruby. They resist the action of most reagents, but the boride SiB_3 is more easily attacked by potash; whilst the compound SiB_5 , richer in boron, is more easily decomposed by concentrated nitric acid. It is curious to compare this simultaneous formation of the two silicon borides, SiB_3 and SiB_5 , with that of the two carbon borides, which are formed in a somewhat similar manner by the action of boron on carbon.

Crystallisation of Gold.—A. Ditte.—If such a metal as gold is placed in contact with the vapours evolved when SO_2 and NaCl react on one another according to the equation $8\text{SO}_2 \text{ gas} + 12\text{NaCl} = 6\text{SO}_4\text{Na}_2 + \text{S}_2\text{Cl}_2 \text{ gas} + 10\text{Cl} + 68.7 \text{ cal.}$, in a porcelain crucible, the whole is heated sufficiently to melt the contained mass of salt and sodium pyrosulphate; the gold is found in a crystalline form, and this at a lower temperature than the fusing-point of the metal. Platinum is attacked in the same way as gold.

Electrolytic Estimation of Bismuth.—Dmitry Balachowsky.—Up to the present time it has not been possible to obtain, by electrolysis salts of bismuth, a sufficiently adherent deposit to allow of washing and weighing. The author has, however, now obtained a deposit of metallic bismuth adhering to the cathode and allowing of washing and quantitative determination. The conditions necessary to obtain this result are—(1) Slight acidity of the solution; (2) absence of large quantities of Cl , Br , I ; (3) not too strong a current (maximum 0.060 ampère); (4) unpolished electrodes. The author's experiments were carried out on sulphate and nitrate of bismuth, but not on the chloride.

Amalgams of Sodium and Potassium.—M.M. Guntz and Féréé.—When sodium is dissolved in mercury and the mercury slowly cooled, beautiful crystals of the amalgam separate out. These are of the cubic form, and correspond to the formula NaHg_6 . If, instead of extracting the crystals from the mercury the mass is compressed in leather by hand, still the same amalgam remains; the liquid part, which is filtered off, is mercury saturated with sodium at the temperature of the experiment. The authors attribute this result to the fact of the compound NaHg_6 being a mixture of two amalgams, Hg_6Na and Hg_3Na . The latter amalgam has been obtained in a pure state. They have shown the existence of four distinct amalgams— Hg_6Na , Hg_6Na , Hg_3Na , Hg_3Na . Potassium gives results which are not quite so defined.

Action of Cyanacetic Ethers containing Substituted Acid Radicles on Diazobenzene Chloride and Tetrazodiphenyl Chloride.—G. Favrel.—In a former paper the author showed that the cyanacetic ethers, when reacting on the bis-diazoic chlorides, furnish compounds to which he has provisionally given the formulae which represent the bodies as hydrazones. He now investigates the behaviour of cyanacetic ethers with regard to acid radicles relative to the diazoic and bis-diazoic chlorides. The experiments have been tried with the following ethers:—Ethyl acetylcyanacetate, ethyl propionylcyanacetate, ethyl isobutyrylcyanacetate, ethyl isovalerylcyanacetate, and ethyl benzoylcyanacetate.

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JUNIOR ASSISTANT in the Department of the WAR OFFICE CHEMIST at Woolwich (20–25), 30th August.

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Particulars of the proposed Amendment were set forth in the "Illustrated Official Journal" (Patents) issued on the 9th of August, 1900.

Any person, or persons, may give notice of opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

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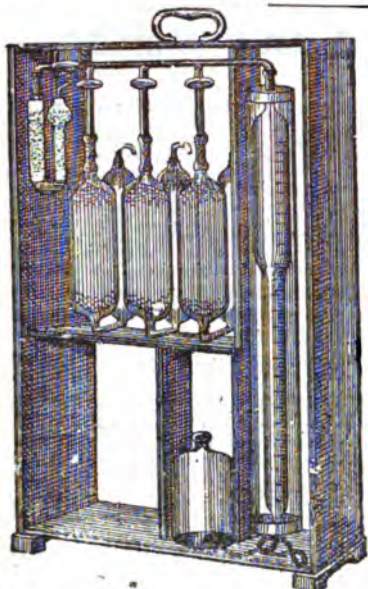
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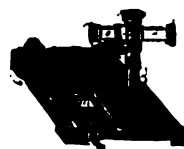
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2126.

ARTIFICIALLY RADIO-ACTIVE BARIUM.

By A. DEBIERNE.

M. AND M^{ME}. CURIE have shown that bodies which remain for some time in contact with radio-active bodies become themselves radio-active, owing to the phenomenon of induction. I have tried to obtain induced radio-activity by means of actinium, and I have especially examined its action on the barium salts.

Bodies rendered active by contact with a radio-active body become very slightly active, and the excited mass is also very small. A stronger induced radio-activity and a greater excited mass can be obtained by a more intimate contact, such as obtaining the active body in solution with the inactive substance, or precipitating the two together.

By this means I have obtained an active barium chloride, by allowing the barium to remain in solution with a very active actinium salt.

Still more intense results can be obtained by precipitating barium sulphate in a solution containing actinium. I have already noticed that, under these conditions, actinium is carried down with the barium sulphate on precipitation. The precipitate is now obtained in the form of chloride, from which the actinium can be separated by precipitation by ammonia in the form of hydrate.

If barium and actinium are left for a short time in contact, the induced activity in the barium is insignificant; if left for a longer time, it is found that the induced activity increases with the time of contact, at least for ten days or so. I thus obtained a considerable quantity of radio-active barium chloride, of activity several hundred times that of ordinary uranium. It was very interesting to discover whether the properties of this artificially radio-active barium are the same as those of radiferous barium extracted from pitchblende.

Certain properties are common to both. The radio-activity of barium rendered active by contact is an atomic property in the same way as that of radiferous barium, since it persists in all chemical transformations.

The rays emitted seem to be similar; they ionise gases, provoke phosphorescence of barium platino-cyanide, and act on photographic plates. Further, a portion of the rays are deviated in the magnetic field, and the anhydrous chloride is spontaneously luminous.

Finally, artificially radio-active barium can be fractionated in the same way as the radiferous chloride. When crystallised from water or hydrochloric solution, the chloride which crystallises is more active than that which remains in solution.

This property, which indicates a difference in solubility between the active and ordinary barium chloride, allows of the concentration of the activity, enabling the preparation of products a thousand times more active than ordinary uranium.

However artificially radio-active barium differs from the barium direct from pitchblende, in the following important properties:—It does not possess the spectrum of radium; M. Demarçay examined a product about a thousand times more active than uranium, and was not able to discover the radium lines, whilst with a product only ten times as active as uranium, extracted from pitchblende, the radium spectrum is decidedly visible.

Another important difference has also been observed. The radio-activity of artificially radio-active barium chloride diminishes with time. In three weeks it has diminished to one-third, whilst the activity of radiferous

barium chloride or salts containing actinium begin by increasing, then remain constant. It is very important to prove that this induced radio-activity is not due to traces of actinium or radium. This explanation is scarcely reasonable, considering the methods of separation used. The absence of a spectrum and the diminution of the activity show that this induced activity is due neither to radium nor to actinium.

It is clear, from the preceding, that I have obtained by induction a radio-active barium, which is clearly distinguished from barium and radium, and which apparently is intermediate between these two elements.—*Comptes Rendus*, cxxxi., No. 5, July 30, 1900.

ON THE THEORY OF THE FORMATION OF SULPHURIC ACID.

By E. LOEW.

THE contents of each chamber in a sulphuric acid plant consist of a gaseous mixture tending towards a state of equilibrium, but, though this equilibrium is never reached, a stable character is, however, produced, characterised for each chamber by the mean conditions of the composition of the gases and the temperature.

To begin with, the manufacture of sulphuric acid is intermittent. When the gas from the pyrites oven fills a certain space at the ordinary temperature, and steam with the oxides of nitrogen are allowed to mix with it, the reaction will be as follows:—The temperature rises to a maximum, then falls to its original point, the proportion of SO₂ present diminishes and disappears. Liquid sulphuric acid is formed, but not with the speed corresponding to the equation SO₂ + O + Aq = H₂SO₄Aq, as the fumes of sulphuric acid only condense slowly. The process of the reaction is uniform throughout the chamber, but the speed of the reaction is very different at the commencement and the finish, as it is a function of the temperature and the concentration of the reacting gases: when the reaction is complete, the chamber is filled with a homogeneous gaseous mixture, though during its progress—owing to inequalities of temperature, &c., at different points—the gaseous mixture may not always be absolutely homogeneous.

The most convenient chamber for the study of the theory of the formation of sulphuric acid would be one constantly filled with a homogeneous gaseous mixture, in which the gas was admitted, and passed out not at one point only, but at an infinite number of points, uniformly throughout the chamber: such a chamber, however, cannot exist.

The law of Guldberg and Waage can, however, be applied to any fraction of a chamber, and the mean gaseous composition calculated therefrom. Admitting that the reaction of the homogeneous mixture takes place at a constant temperature, the law of masses is applicable.

The general equation of the reaction is:—



where A represents the kind, and *n* the number of molecules. Let *c*₁ *c*₂ *c*₃ ... be the number of grm.-molecules of the substances A₁ A₂ A₃ ... per litre, then the speed V of the reaction is:—

$$V = v - v' = Kc_1c_2c_3 \dots - K'c'_1c'_2c'_3 \dots$$

(K and K' being constants of proportion),—that is to say, the speed of the reaction produced in the direction of the equation (from left to right) is proportional to the production of the active matter. The compounds reacting are SO₂, H₂O, O₂, N₂O₃(NO). If the reaction leads to nitrosyl sulphuric acid, it may very well be reciprocal. Manufacturers agree that between 50–60° is the best temperature, at which the decomposition of the nitrosyl sulphuric acid is still slight, but, on the other hand, when the temper-

ature rises, $v' = K c_1 c_2 c_3 \dots$ acquires a value nearer to $v = K c_1 c_2 c_3 \dots$, and finally $v' = v$,—that is to say, that the system is in equilibrium, and no more sulphuric acid is formed. These suppositions, however, have yet to be proved. Experiments have shown that the relations of concentration are a measure of the speed of the reaction; it remains to be seen whether it is really according to the formula—

$$V = v - v' = K c_1 c_2 c_3 \dots - K' c'_1 c'_2 c'_3 \dots$$

This has not yet been confirmed.

An excessively energetic reaction has been observed in the first part of the chamber, and a stoppage of the reaction in the second part. I propose to show that this observation is erroneous, and that the second part of the chamber also takes part in the reaction, according to the composition of the gases it contains.

Lunge's conclusions, drawn from the proportion of SO_2 present, can be equally well deduced from the oxygen.

A glance at the analyses reveals figures which may be explained, but which cannot logically be used for explaining the course of the reaction. According to the proportion of SO_2 , Lunge calculates for different parts of the chambers, the progress of the reaction as follows:—

Where the sample for analysis was taken from.	First chamber, beginning.	First chamber, middle.	Junction of first and second chambers.	Second chamber, middle.	Junction of second and third chambers.	Third chamber, exit.
Calculated by Lunge and Naef	57.7	78.15	80.1			
Calculated from the proportion of O_2	54.1	72	80.9			
Lunge and Naef	—	74.8	77.47	94.52	95.45	99.6
Calculated from the O_2	—	67	72	90.6	95.5	100
Lunge and Naef	48.63	59.23	73.9	96.84	97.13	99.3
Calculated from the O_2	65.4	73.7	84.2	100	100	100

It is necessary to find an explanation why the reactions almost entirely cease in the second part of the chamber, and then re-commence energetically in the next chamber. The gases arrive from the furnace full of activity, and both chemical and mechanical reactions take place with energy. It is this violent mixture of the gases which makes a system composed of a single chamber irrational, as it is impossible to maintain a high concentration if the escaping gas is to be free from SO_2 . The greater the quantity of gases in the chambers, compared with the amount of gas coming from the furnace per unit of time, the more equal will their composition keep; also, the more energetic the movement of the gases, and the more the differences produced by the chemical reactions become equalised, the more uniform will the composition of the system be. It remains to be proved experimentally whether the gases become mixed throughout the chamber, and whether the reaction follows the concentration of the gases, as different authors do not agree on this point.

If we suddenly add nitric acid to the contents of the lead chamber, the temperature instantly rises in the first part of the chamber, but in a few minutes the rise is noticed at the further end. In a few more minutes the nitric acid commences to act at the further end. According to different methods of manufacture, the gas takes from two to four hours to traverse the system. The actual direction and speed of the gas is of no importance. In a system of chambers receiving a sudden excess of 4 kilos. of nitric acid through the Glover tower, the following observations were made:—

	At 6 metres from the first side we observed— °C.	At 30 metres from the first side we observed— °C.
Normal progress	56	53
On addition of HNO_3	56.0	53.0
After 2 minutes	57.0	54.0
" 5 "	60.0	56.0
" 6 "	61.0	58.0
" 9 "	61.5	59.0
" 10 "	62.0	60.0
" 15 "	64.0	61.0
" 20 "	65.0	60.5
" 25 "	66.0	59.5

This can only be explained by admitting that a part of the gases from the furnace had a speed considerably above the mean,—that is to say, that the gases were mixed throughout the chamber. The gases mix with difficulty; but when they reach a part of the chamber where the temperature is distinctly different and chemical reactions take place, and an external cooling causes the formation of currents, the conditions for mixing are apparent.

The chemical reactions which take place in the lead chambers are governed by the temperature and the concentration of the reacting molecules. Each part of the chamber has an effect proportional to its concentration. We will now consider the relations of these concentrations.

1. *Steam*.—The maximum quantity of steam which can exist in a lead chamber depends on the vapour-tension of the sulphuric acid formed at the local temperature. If the temperature be 50° , and the strength of the acid 5°B. , the vapour-tension is 10.9 m.m. according to Sorrel, and the density—

$$d = \frac{5}{8} \times \frac{0.00129}{1 + 0.003665 t} \times \frac{\phi}{760} = 0.00001.$$

One cubic metre of gas contains 10 grms. of steam. To form 153 grms. of acid at 51°B. , 73 grms. H_2O and 64 grms. SO_2 are required. The chamber thus contains the amount of water equivalent to 8.77 grms. SO_2 per cubic metre,—that is, 3.05 litres at 0° , or 3.6 litres at the temperature of the chamber. If we keep the acid at 49.7°B. , and the temperature at 60° , the corresponding quantity of water is equal to 17.2 grms. SO_2 per cubic metre, or 0.73 vol. per cent. Chambers which contain too much sulphurous acid are always deficient in water. The necessary amount of steam can only be introduced at one point, but, as the vapours mix rapidly, this condition is favourable, but we must not be too sure that this is the best. It is necessary to introduce water by as many orifices as possible in chambers where the proportion of SO_2 is high; we should therefore expect to get an acid of much greater dilution.

2. *Oxides of Nitrogen*.—The addition of oxides of nitrogen may be increased to a certain extent, but it is practical considerations only which fix the limit. The high production of French chambers necessitates a high consumption of nitric acid. It is not possible to increase the production of a chamber by increasing the proportion of any of the other bodies which enter into the reaction. It should be taken as proved that it is not the relation of the concentrations that determines the speed of the reaction. The chambers use more SO_2 in winter than in summer, and for a plant of 3000 cubic metres (3 chambers) this increase reaches 15 per cent; the consumption of oxides of nitrogen, calculated to nitric acid at 36°B. , is from 0.9 to 1.1 per cent in summer, and from 0.93 to 1.2 per cent in winter, of the quantity of sulphuric acid at 60°B. obtained. The temperature of the first two chambers is practically the same in summer and winter. We can therefore only attribute this increased production in winter to the greater cooling of the sides and the better condensation of the sulphuric acid fumes. There is no reason to doubt that the reaction takes place as described by Lunge. If the oxides of nitrogen become fixed in the state of nitrosyl sulphuric acid, the proportion of gas in

one of the reacting bodies diminishes, and the reactions which tend to destroy the compound formed ought therefore to play a part in the formation of the sulphuric acid. The speculations of Sorrel on the vapour tension of nitric acid in the presence of sulphuric acid throw a great deal of light on these phenomena.

3. *Oxygen*.—The proportion of oxygen varies within certain limits, according to the amount of SO_2 in the gas from the pyrites furnace.

4. *Sulphurous Acid*.—From what we have said above, the higher limit of the proportion of sulphurous acid can be determined. The relation between the proportions of O_2 and SO_2 is not to be neglected, according to the Guldberg-Waage law; it can be calculated whether it is more advantageous, for example, to work with the furnace giving a gas richer in oxygen and poorer in sulphurous acid, or the contrary. As a general rule, the average proportion in SO_2 of the chamber-gases is lower than that of the furnace-gases. We determine the mean proportion to be kept up in each chamber, knowing that this chamber ought to supply the next with a given quantity of gas of such proportions that the following chambers can complete the reaction. It is evident, therefore, that a plant consisting of a single chamber will give a very poor return.

We must consider a definite concentration of SO_2 , N_2 , O_2 , H_2O , and O , for certain conditions, as the normal concentration; a variation from this standard leads to an abnormal condition, and it must be remembered that, under certain circumstances, the contents of the first chamber of a system may undergo rapid variations, which, however, are of short duration, and are hardly felt in the following chambers; once the normal conditions are re-established in the first chamber, the proper concentrations remain a long while in the second. The analyses of the gases may sometimes lead to confusion; the second chamber may show over production while analysis shows the contrary; we find, for example, x per cent of SO_2 entering and y per cent leaving, while actually the chamber may have consumed a larger proportion of SO_2 than has entered, and has only lost a part of the abnormal quantity it contained; this, however, is an extreme case.

To sum up it may be said:—

1. The chemical reactions giving rise to the formation of sulphuric acid follow the Guldberg-Waage law, each part of the chamber giving a production corresponding to its concentration in reacting matter.

2. The mixture of the gases is complete, and the composition of the contents of the two halves of the chamber practically identical.

3. The proportion of O_2 to SO_2 is constant; it varies within certain limits according to the size of the chamber.

4. By adding an excess of oxides of nitrogen, and by creating conditions by which a greater proportion of steam can be kept up, the speed of the reaction in the chambers can be increased.

5. The difference in temperature of two chambers is in strict relation with the manner in which the reaction $\text{SO}_2 + \text{O} = \text{SO}_3$ is divided between them.—*Zeitschrift für Angewandte Chemie*, April 3, 1900.

Combination of Sulphate of Nickel with Hydroxylamine.—R. Uhlenbuth. When we add anhydrous hydroxylamine to a cold saturated solution of SO_4Ni , the green colour of this salt gives place to an azure blue, and finally to a deep red tint. The solution then slowly (or rapidly on the addition of a little alcohol) deposits red crystals, probably quadratic, of the salt $\text{SO}_4\text{Ni} + 6\text{NH}_2\text{OH}$ (instead of $6\text{H}_2\text{O}$), which consists of the hydrated crystals. These crystals are decomposed by water and by alcohol, with the separation of a white powder which has not yet been examined. On heating the red salt it swells up and changes colour, becoming successively blue and then green. The blue salt, without doubt, contains less hydroxylamine.—*Lieb. Ann. Ch.*, vol. cccvii., p. 332.

THE GASOMETRIC MEASUREMENT OF NITRITES IN THE PRESENCE OF NITRATES OR OTHER SOLUBLE SALTS.

By J. GAILHAT.

HAVING had occasion to estimate the proportion of nitrites contained in various saline mixtures during a research on the nitroprussiates, we were led to the conclusion that among the various methods recommended for this purpose but few possess the precise and practical qualities necessary for obtaining good results.

The use of the manganimetric method, not to mention others, is very much restrained by reason of a large number of substances, as many mineral as organic, which act on the permanganate under the conditions of the experiment.

The following process has the advantage of being available under widely different conditions, while all the time retaining its absolute exactitude.

When, to an excess of a neutral, boiling, concentrated solution of sal-ammoniac, we add a solution of a metallic nitrite also neutral, we get a regular disengagement of nitrogen according to the following formula:—



The equation shows us that to get at the quantity of nitrite engaged in the reaction, it suffices to determine either the amount of sal-ammoniac decomposed, the nitrogen given off, or the fixed chloride produced. Practically, however, the nitrogen gas is the most readily adapted to a simple and rapid experimental estimation; it can easily be collected and accurately measured.

The apparatus we have made use of is the same as that used by M. Schlösing for the gasometric estimation of the nitrates. We have adapted to it an escape tube formed in a single piece, and fitted with a tap which greatly facilitates the operation. Further, the water-jacket is deep enough to allow of the complete immersion of the tubes graduated to tenths of a c.c. in which the nitrogen gas is collected. Finally, we constantly maintain a current of water through this jacket sufficient to prevent any rise of temperature.

Thanks to these precautions, the cooling of the water contained in the tubes being assured, the gases held in solution are unable to come off, and the nitrogen collected is also unable to be dissolved therein, as this water is already saturated.

To conduct an operation the flask is filled to about two-thirds of its volume with a saturated solution of chloride of ammonium; the apparatus is set up, and the temperature brought to boiling-point, with the taps remaining open. The pressure causes the liquid in the funnel tube to rise, and the tap is turned as soon as the liquid reaches its level. The boiling is allowed to proceed until all the air is driven off. We can make sure of its complete expulsion by closing the tap of the escape tube for a moment; the water in the water-jacket then rises up to this tap, making a sharp little noise. If, at this moment, any gas remained in the tube, the quick opening of this same tap would easily allow it to be driven out on account of the increase of pressure produced in the flask during this little manoeuvre, which may be repeated two or three times to make sure of the complete absence of any trace of air. We then place in the funnel 10 c.c. of solution of the nitrite to be estimated, of which the approximate amount present should be between 5 and 10 grms. per litre; the graduated tube is placed at the end of the escape tube, and kept slightly inclined at the bottom of the water-jacket; the nitrous solution is then allowed to run in, taking the necessary precautions for preventing any absorption. The rapid, regular disengagement of gas soon commences; the funnel is rinsed two or three times, and the nitrogen allowed to come off completely. This can be verified as shown above.

All the readings are made at the same moment, noting the temperature, the barometric pressure, and the vapour tension of the water under these conditions. Thus we have all the elements for the calculation. The vapour X, the weight of nitrite contained in a litre of the solution under examination, being given by the expression:—

$$X = \frac{100 M}{14} \times \frac{V}{2000} \times \frac{H-f}{(1+a t) \times 760} \times 0.9712 \times 1.293,$$

in which M represents the molecular weight, or rather the equivalent of the nitrite estimated, and V the volume of nitrogen obtained expressed in cubic centimetres, the half of which only comes from the nitrite, the remainder being due to the decomposed sal-ammoniac.

We have checked the accuracy of this method by means of solutions of nitrite of potassium purified with alcohol, taking varying quantities, then weighing rapidly after fusing the pure salt and cooling in an exsiccator, as well as by estimations made by the manganimetric method with solutions of nitrite of potassium and nitrite of sodium.

	I.	II.	III.	IV.
Expt. made with 10 c.c. of a solution of—	NO ₂ K	NO ₂ K	NO ₂ Na	NO ₂ Na
Containing per litre, Grms.	9.81	5.94	—	—
Value of V, C.c. ..	1. 26.72	18.48	20.75	26.20
	2. 26.70	18.52	20.80	26.25
	3. 26.80	18.50	20.82	26.25
	4. 26.75	18.54	20.80	26.28
	5. 26.73	—	—	26.23

Corresponding value of X,	Grms.	9.84	5.97	5.32	7.57
Result given by the	1.	9.74	5.80	5.28	7.58
MnO ₄ K method..	2.	9.78	5.86	5.31	7.64

The agreement is perfect; the process may be used, in most cases, where the salt to be estimated is in the form of a more or less complex mixture.

We can also operate comparatively, in a more simple manner, with a typical solution of nitrite obtained from a pure salt, and weighed or titrated colorimetrically.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xii., No. 1.

REFRACTIVE INDEX AND ALCOHOL-SOLVENT POWER OF A NUMBER OF CLEARING AND MOUNTING MEDIA.*

By C. E. M'CLUNG.

As far as the author's observation goes, there has not been published any very extensive list of the refractive index and clearing value of the reagents commonly used by microscopists. Such a want is noted by Lee in the fourth edition of his "Vade Mecum," page 66. In the hope of assisting in the compilation of such valuable information, the writer has experimented on the reagents named in the following table, with the results there noted. The instrument employed in the determination of the refractive index was the Pulfrich refractometer, the sodium flame being used as the source of illumination and the conditions of temperature being kept as nearly constant as possible.

In ascertaining the clearing value of the substances, a number of methods were tried, but finally abandoned for the simple one of testing the strength of alcohol that would dissolve in the reagent and produce a clear solution. While this method does not truly represent the conditions present in a tissue where the clearing agent is replacing the absorbed alcohol, yet actual practice indicates that the results are approximately correct, at least nearly enough so to make the figures of practical value. Strengths of alcohol varying by 5 per cent were employed, and the weakest one that the reagent would dissolve is given in the table as the lowest grade of alcohol from which it will clear.

* From the *Kan. Univ. Quarterly*, vii., No. 4.

The different substances tested were taken from the ordinary laboratory supply, and supplemented by others obtained from the Pharmacy department of the University. The latter consisted almost entirely of essential oils from the house of Lehn and Fink, New York. When possible, more than one sample of each reagent was obtained and tested.

Following is the table:—

	Refractive index.	Strength of alcohol dissolved. Per cent.	Price per pound. Dollars.
Chloroform	1.4395	95	0.65
Linalool	1.45941	80	—
Oil, Eucalyptus ..	1.46090	90	1.25
Linaloe	1.46090	85	—
Oil, Petitgrains ..	1.46090	90	—
" Coriander	1.46288	85	0.75 oz.
" Peppermint	1.46327	85	1.25
" Cedar	1.46626	95	0.30
" Pinus sylvestris ..	1.46882	100	1.10
Turpentine	1.46882	100	1.40 gal.
Oil, Lemon	1.47078	95	1.25
" Eucalyptus glob..	1.47097	90	—
" Orange	1.47176	95	4.00
" Pinus picea	1.47274	100	—
" Juniper berries ..	1.47394	95	1.75
" Pinus pimplionis ..	1.47620	100	—
" Citronella	1.47909	90	1.25
" Origanum	1.47919	95	1.10
Turpeneol	1.48005	75	—
Oil, Celery	1.48054	95	1.20
" Nutmeg	1.48054	95	0.30 oz.
" Origanum	1.48103	95	1.10
" Caraway	1.48441	90	1.75
" Ginger	1.48807	95	0.65 oz.
Xylene	1.49348	95	—
Benzene	1.49488	95	0.45
Carvol	1.49535	85	—
Oil, Thyme	1.49638	95	1.49
" Copaiva	1.49723	Immisc.	1.10
" Cedarwood	1.50188	95	—
"	1.50326	95	—
" Cumin	1.50373	90	4.50
" Sandalwood (E. In.)	1.50510	90	6.00
" Calamus	1.50602	95	0.30 oz.
" Sandalwood (W. In.)	1.50820	80	3.50
" Cedarwood	1.51533	95	—
Xylene balsam ..	1.52397	—	—
Oil, Sassafras	1.52724	95	0.50
" Allspice	1.53062	85	0.25 oz.
" Cloves	1.53171	80	0.65
" Sweet birch	1.53329	90	—
" Cinnamon leaves ..	1.53535	85	—
Safron (sp. gr. 1.108) ..	1.53584	95	—
Oil, Fennel	1.53885	95	1.75
" Cloves	1.53723	80	0.65
" Mirbane	1.54982	95	0.35
" Anise	1.55795	95	2.25
Anethol	1.55208	90	—
Anilin	1.58457	60	—
Oil, Cassia	1.60160	80	1.80

The table speaks for itself, so that comment is scarcely necessary. From the list given, a series of clearing agents may be selected in which almost any refractive index from 1.44 up to 1.60 is obtainable. Since many of these are suitable agents in which to mount objects, the proper refractive power for most structures may be selected. Where it could be found, the price of the reagent per pound is given.

Particular attention is called to the value of the oil of cassia, which has a refractive index of 1.60160 and clears from 80 per cent alcohol. It is a most excellent reagent according to all experience in the laboratory, and in addition to its other good qualities, it dries hard enough to make permanent mounts.

SYNTHETIC INDIGO.*

By EDWARD S. JOHNSON.

It is a matter of very general observation that in this centre of occidental civilisation, the United States, the reception accorded scientific discovery by the mass of intelligent men is notably apathetic unless prospect for immediate utilisation is evident. This fact is pointed to as a marked characteristic of the American by the enlightened nations of the Old World, where, pre-eminently in Germany, the greatest importance is attached to scientific research and discovery whatever the momentary outcome.

The causes of these divergent social conditions cannot consistently be made here a subject of minute discussion. It suffices to refer to the influence of environment and other factors of natural selection in their bearing upon the older civilised nations treated purely as modified varieties or races of the *genus homo*, and the intensity, not to say fierceness, of the struggle for existence occasioned by the narrow limits naturally prescribed for a multitude of combatants. Such conditions have resulted in resort to all the refinements of human intelligence to maintain a successful struggle; in all branches of industrial affairs, aid has been drawn from institutions of learning with such apparent advantageous effect as to establish in those countries where scientific light is brightest an intimate connection with, and supreme respect for, science in its purity and the increasing host of its devotees.

Cultured young America has not been altogether backward in an appreciation of such matters in what has been his absorbing occupation, the amassing of wealth. The untold natural resources of a new land he has in part brought to light, utilised to his own comfort and set his sons of the last decades of the century upon a career of development unparalleled in rapidity and immensity to a degree which makes America an object of utter amazement to the more cultured of older civilisations. With this growth has come a severity of competition in all those forms of great organised effort of civilised man, known in part in social economics as industry and commerce. Natural science is exerting in all departments of industry an elevating influence. The united efforts of botanist, zoologist, and chemist are fast placing agriculture upon a rational footing, and those manufacturing industries producing the great staples which serve as raw materials for the almost innumerable objects of cultured necessity and comfort are directed each year more fully than ever by the deductions of physical and chemical science.

Many stages of progress must, however, still be attained and passed before American industry may generally be regarded as upon the level of that of the leading nations of continental Europe in respect to an appreciation and application of scientific methods and observations. That period of development has hardly passed in which a proffered advantage is eagerly accepted, fairly grasped by the beneficiary, while the benefactor is relegated to forgetfulness, or, if remembered, referred to with a kindly commiserative, amused smile, when engaged upon problems of science not promising an instant contribution to material wealth. Leaving the figure of the youth in the first years of robust manhood, with fine muscular development and general functions, but with an indefinite sense of the source of his well-being, American industrial conditions may be again likened, in a measure at least, to those of the newly discovered gold-fields. Methods of operating are adapted to a dazzling natural wealth of raw material to be converted into finished product by a mere turn of the hand, for the very picking up, or, at most, the crudest forms of placer mining. The mass of fabulous treasure contained beneath the surface in great alluvial deposits and beds of quartz rock is neglected for the time, and left for

the systematic and refined operations of the metallurgist, who in after years applies the knowledge of his science with the utmost economy in the transformation of his raw materials. This latter period of the gold field's history illustrates in main features European industrial conditions.

Nothing offers a more forceful illustration of the important part which the cultivation of science, for its own sake, has played in their development to the present exalted level than the following, at first glance apparently insignificant incident, from the history of synthetic indigo.

In pursuance of confirmation of his theory of the relationship of indol to the compounds of the indigo-group, Baeyer (*Ber. d. Deutsch. Chem. Ges.*, xlii., 2254; *Ueber d. Beziehungen d. Zimmtsaure zu d. Indigogruppe*) sought evidence in the reduction of isatine to diosindol, oxindol, and finally indol. Success was ultimately achieved, but not without the expenditure of, seemingly, a wasteful amount of time and energy to discover the right reducing agent. This was found in the now classic, although common, zinc-dust of the laboratories, which has been, and is, of greatest service to the practice of chemical research, and literally has led to the establishment of a gigantic chemical industry. It was by the application of the new reducing agent that Graebe and Liebermann, respectively assistant and pupil in Baeyer's laboratory, in 1868, discovered the constitutional relation of alizarine to anthraquinone and anthracene, and soon were led to invent means for a synthesis of the celebrated colouring-matter which, authentic accounts relate, produced the flaming reds of the Far East centuries and centuries ago.

The cultivation of the madder plant, the source of this ancient dye-stuff, gradually kept westward, and it is related (Schultz, *Chem. d. Steinkohlentheers*, ii., 584) that, as early as the seventh century, the plant was grown on a small scale in France, which became, however, several centuries later, with favourable climatic conditions, the most extensive European producer of madder, the ground root of the colour-bearing plant. Besides France, Holland engaged notably in the cultivation. In the season of 1862-3, the production (Schultz, *Chem. d. Steinkohlentheers*, ii., 597) of madder in France had attained the seemly proportions of 26,850 tons; ten years later, 1871-2, and two years after the introduction of artificial alizarine, 25,000 tons were still harvested; in 1878-9 this sum had decreased, gradually at first, and then by leaps and bounds, to 500 tons. A similar, but less rapid, fate overtook the production in other madder-growing lands, dwindling the estimated total output of the root, 70,000 tons, for the years about 1868 to a mere trifle. The rapid decline in the cultivation was accompanied, which was still more disastrous to the madder-grower, by a fall of 75 per cent in price. This is concretely exhibited by the fact that in 1860 the value of madder and garancine exported by France was nearly 6,250,000 dols., an amount which had decreased, for the same items in 1876, to somewhat more than 900,000 dols.

By these industrial changes, no mean crisis had presented itself. Vast tracts of valuable agricultural land must be turned to lucrative account, and extensive commercial organisations must be dissolved or seek excuse for existence in other fields. While a temporary disturbance was occasioned, the general effect can only be regarded as beneficent; the development of the great staples of agriculture was elevated where such was urgently needed by the return of former madder-fields to their cultivation. It is most interesting to note the influence of the innovation upon chemical industry, the facts of which relate a familiar story, but one of such cardinal character as to bear frequent recounting, marking, as it does, an epoch in the history of applied chemistry. By the announcing of a practicable manufacture of alizarine from anthracene, the preparation of the latter became one of the most important and lucrative operations of the coal-tar distilleries. For the conversion of anthracene into anthraquinone, a heavy demand was made upon the

* Proceedings of Engineers' Society of Western Pennsylvania, xvi., No. 3.

producers of the alkali chromates. In succeeding transformations, the sulphonation and oxidising fusion, fuming sulphuric acid, caustic alkali, and potassium chlorate are required. Their supply by the acid, alkali, and chlorate industries gave each a strong forward impulse, adding heavily to the production.

This splendid industrial expansion, which the enthusiastic chemist cannot regard but with keen pleasure and pride, may be directly referred for its origin to the simple observation of the value of zinc dust in the study of a group of substances belonging in a totally different field of scientific research from that by which by its means was later illumined and cultivated with marvellous results. The influence of this reagent as an industrial factor did not cease with the founding of the alizarine manufacture. Its part in revealing, in the hands of Baeyer, the relationships of indigo, and leading thus finally, in the course of years and a series of researches of unequalled brilliancy, to practicable methods for its synthesis, marks the distant beginning of another economic change now steadily progressing toward certain successful culmination.

(To be continued).

ON THE EFFECT OF VERY LOW TEMPERATURES ON THE COLOUR OF COMPOUNDS OF BROMINE AND IODINE.

By J. H. KASTLE.

SOME time ago the writer called attention to the fact that the characteristic red, orange, or yellow colour of bromine and iodine compounds could probably be accounted for on the supposition that the halogen compounds exhibiting such colours are slightly dissociated even in the solid state, and that the characteristic colour, therefore, is simply that of the halogen itself. The following facts were cited in support of this conclusion:—

1. The colour intensity of the halogens is in inverse ratio to their chemical activity, and may be represented roughly at least by $F < I$. Similarly, the colour intensity of their compounds by $MF < MI$. Further, the colour of bromine and iodine compounds is altogether similar to that exhibited by certain solutions of these two elements.

2. The perfect continuity exhibited in the colour changes of such easily dissociable substances as phosphorus pentabromide in passing through the solid, liquid, and gaseous states.

3. Those halogen compounds are the most highly coloured which are the least stable, and whose elements are held in combination by the weakest affinities.

4. On heating, the colour of halogen compounds becomes darker and deeper in tint.

It also follows that, if the characteristic colour of bromine and iodine compounds is due to dissociation, the colour of these compounds ought to become lighter in tint, if not altogether white, on cooling to very low temperatures. At the time of my first communication on this subject, no opportunity was afforded for trying the effect of very low temperatures on the colour of the compounds in question. Through the kindness of Dr. Freer, of the University of Michigan, and Dr. Simon, of the College of Physicians and Surgeons, Baltimore, I have recently been able to try the effect of very low temperatures (the boiling-point of liquid air -190°) on the colour of certain of these halogen compounds. On the 9th of March Dr. Freer lectured on liquid air in the city of Louisville, and on the 22nd of March Dr. Simon lectured in Cincinnati on the same subject. At the close of the lecture both of these gentlemen kindly placed at my disposal a quantity of the liquid. The following compounds were selected for experiment:—Lead iodide, phosphorus pentabromide, phosphorus heptabromide, mercuric

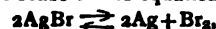
iodide,* iodoform, mercuric bromide, benzenedibromsulphonamide, and tribromphenol bromide. Small amounts of these compounds in pure condition were placed in sealed tubes. These were then immersed in liquid air in a Dewar tube, and allowed to remain in the liquid until no further alteration of colour was observable. The colour of these compounds before and after cooling in liquid air was observed to be as follows:—

Substance.	Colour at ordinary temperatures.	Colour at the temperature of liquid air.
Lead iodide . . .	Golden yellow..	Pale sulphur-yellow.
Phosphorus pentabromide . . .	Citron yellow ..	White or very pale yellow.
Phosphorus heptabromide (a) . . .	Red	Yellow.
Mercuric bromide . . .	Pale yellow ..	White.
Iodoform . . .	Yellow ..	White, or nearly so.
Benzene dibromsulphonamide ..	Orange ..	Pale sulphur-yellow.
Tribromphenol bromide . . .	Yellow ..	Very pale yellow.
Mercuric iodide ..	Red ..	Orange yellow.

(a) This compound has hitherto been regarded as the red modification of phosphorus pentabromide. Kastle and Beatty have recently shown, however, that the substance is in reality a higher bromide of phosphorus, probably the heptabromide, PBr_7 .

It will be observed that all of these compounds became markedly lighter in colour in cooling to $-190^\circ C.$, and in some cases the change of colour was most striking. It would seem, therefore, that these results tend to confirm the idea that the colour of halogen compounds is due to dissociation.

Before leaving this phase of the subject, it might be well to call attention to an observation by van't Hoff. In his "Etudes de Dynamique Chimique" (Eng. Trans., p. 273) he points out that at $60^\circ C.$, *in vacuo*, silver bromide dissociates in the sense of the equation—



and that silver bromide at 60° *in vacuo* will decompose until the bromine vapour evolved has reached a pressure of 2.9×10^{-11} m.m.

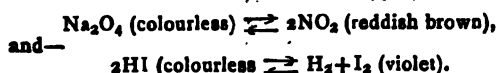
It would seem to be generally, if not universally, true that coloured substances become lighter in colour on cooling to very low temperatures. That such is the case may be seen from the following:—

Substance.	Colour at ordinary temperature.	Colour at -190° .
Bromine . . .	Dark red, opaque	Orange-red.
Iodine, solid . . .	Steel-grey ..	Steel-grey.
Iodine, in alcoholic solution . . .	Brown ..	Very light yellowish brown.
Iodine, in carbon disulphide solution . . .	Pink	Very light pink.
Sulphur, rhombic.	Sulphur yellow.	Nearly colourless.
Phosphorus, red modification . . .	Dark crimson ..	Ferruginous brown
Copper sulphate..	Dark blue..	Dark blue.
Chromic alum . . .	Dark plum colour	Pinkish violet.
Manganous chloride . . .	Pink	Very light pink.
Chromic chloride.	Violet	Pink.
Phenolphthalein (alcoholic solution, alkaline) ..	Pink	Colourless.
p-Nitrophenol (alcoholic solution, alkaline . . .	Yellow ..	Lighter yellow.

* Other observers have found this compound to become yellow in boiling oxygen.

At the temperature of liquid air fluorescein and eosin in alkaline alcoholic solutions retained their characteristic colours, but became considerably lighter in tint.

From these few observations it would scarcely be logical to conclude that the lightening of colour produced by cooling to very low temperatures is in all cases due to the same cause as that which operates in the case of bromides and iodides. On the other hand, it is an interesting and suggestive fact that any increase in the depth of colour produced by rise of temperature is usually accompanied by a corresponding increase in the chemical activity of the substance, and with such facts before one it is almost impossible to keep out of mind such reversible processes as the following:—



In the light of the electrolytic dissociation theory regarding the action of indicators, the effect of very low temperatures on the colour of an alcoholic solution of phenolphthalein which has been rendered alkaline with caustic soda, is certainly most remarkable and interesting.

There is still another point of interest connected with this subject. This is regarding the effect of very low temperatures on the colour of allotropic modifications of the same substance. It is well known that the red modification of mercuric iodide becomes orange-yellow when cooled in liquid oxygen, and that on removal from the bath of liquid oxygen it quickly changes to red again. By some chemists this has been construed to mean that at very low temperatures the yellow variety of this compound is more stable than at ordinary temperature, and that the yellow form of the iodide obtained by great cooling is identical with that produced by heating above 128° (Newth's "Inorganic Chemistry," 5th edition, p. 558). There can be little doubt regarding the correctness of the first of these conclusions, namely, that the yellow modification of mercuric iodide is more stable at these very low than at ordinary temperatures. It has been shown, for example, that at -35° monoclinic sulphur changes to orthorhombic about five hundred times more slowly than at ordinary temperature (See also "The Phase Rule," Bancroft, 32-34). To conclude, however, that the yellow form of mercuric iodide obtained by cooling the red to -190° C. is identical with the yellow variety obtained by heating the red to 128° is certainly incorrect, as may be seen from the following:—

Small amounts of mercuric iodide were sublimed in test-tubes. These tubes were then allowed to stand until a certain amount of the yellow had changed to red. Both the red and yellow varieties of this compound were thus obtained side by side in the same tube. On placing such tubes in liquid air, it was observed repeatedly that the red variety of the compound became orange-yellow, and that the yellow variety became white or very pale yellow. Above the level of the liquid air the yellow modification of the compound retained its ordinary yellow colour, thereby rendering these differences in colour the more pronounced by contrast. The difference in colour between the red and yellow modifications of mercuric iodide at -190° C. was thus rendered plainly visible. The effect, therefore, of these very low temperatures on the red modification of mercuric iodide is not to convert it into the yellow variety stable above 128°, but simply to lighten its colour in a manner characteristic of other coloured bromides and iodides. Therefore, at all temperatures below 128°, the red and yellow modifications of mercuric iodide are distinctly different substances.

In conclusion, I desire to thank Professors Freer and Simon for their kindness in furnishing the liquid air necessary for these experiments.—*American Chemical Journal*, xliii., No. 6.

THE ACTION OF AMMONIUM CHLORIDE UPON NATROLITE, SCOLECITE, PREHNITE, AND PECTOLITE.*

By F. W. CLARKE and GEORGE STEIGER.

In our last paper upon the ammonium chloride reaction (*Am. J. Sci.*, Feb., 1900; *CHEM. NEWS*, lxxxi., 187), we showed that analcite and leucite, when heated to 350° with this reagent in a sealed tube, both yielded the same compound, ammonium leucite, $\text{NH}_4\text{AlSi}_2\text{O}_6$. We also showed that the reaction was not limited to these minerals, but that it was fairly general in character, and that with other species analogous results could be obtained. We now have data relative to four more minerals, and these exhibit a range of variation which well illustrates the availability of the method for investigations into the chemical constitution of silicates. Three of the species now studied have previously been regarded by one of us as analogous in structure, provided that all or part of their water can be interpreted as constitutional, and the formulæ assigned were as follows:—

Scolecite	$\text{Al}_2(\text{SiO}_4)_3\text{CaH}_4\cdot\text{H}_2\text{O}$
Natrolite	$\text{Al}_2(\text{SiO}_4)_3\text{Na}_2\text{H}_4$
Prehnite	$\text{Al}_2(\text{SiO}_4)_3\text{Ca}_2\text{H}_2$

Two of these formulæ must now be abandoned, because of the experimental evidence which we have obtained. We may first study the three species individually.

Natrolite.

In our former paper we reported a crude, preliminary experiment made upon impure, yellowish natrolite from Aussig in Bohemia. After heating with ammonium chloride in a sealed tube, and subsequent leaching with water, 17.56 per cent of soda was extracted, and in the residue 8.29 per cent of ammonia was found. Natrolite, therefore, was a suitable mineral for further investigation, and our expectations regarding it have been fully confirmed.

The material available for our new experiments came from the well known locality at Bergen Hill, New Jersey, and consisted of a mass of slender needles densely matted together. Part of the uniform, ground sample was analysed, with fractional determinations of the water, and part was used for the sealed tube experiments, precisely as in the research upon analcite and leucite. Three of these experiments were made, and in each case the natrolite was mixed by grinding in an agate mortar with four times its weight of dry ammonium chloride, after which it was heated to 350° in the sealed tube. Even during the grinding a slight reaction took place, and a distinct smell of ammonia was given off by the mixture. With pectolite the same smell was perceived. The three experiments may be summarised as follows:—

- Heated eleven hours. Upon leaching, 14.89 per cent of soda and 1.20 of lime were extracted. In the residue 9.26 per cent of ammonia was found.
- Heated nine hours. Leach not examined. 9.26 of ammonia in residue. The complete analysis of the residue is given further on.
- Heated three hours. 14.09 per cent of soda and 0.20 of lime were extracted. The residue contained 8.87 per cent of ammonia. In this instance the heating was relatively brief, in order to learn whether its duration could be advantageously lessened. The reaction was evidently less complete than in experiments A and B.

In the subjoined table we give first the analysis of the natrolite itself, and then that of the leached residue from experiment B. In the latter we found that 0.86 per cent of silica was soluble in sodium carbonate solution, and that soda and lime remained corresponding to 4.61 per

cent of the original mineral. Deducting these impurities, together with the 0.42 per cent of hygroscopic water, and re-calculating to 100 per cent, we get the *reduced* composition of the residue. In the last column is given the calculated composition of an anhydrous ammonium-natrolite, $(\text{NH}_4)_2\text{Al}_2\text{Si}_2\text{O}_{10}$. This compound has evidently been formed to an extent represented by over 94 per cent of the leached natrolite residue. The agreement between theory, and even the un-reduced analysis, is practically conclusive on this point.

	Natrolite found.	Residue reduced.	Residue $(\text{NH}_4)_2\text{Al}_2\text{Si}_2\text{O}_{10}$ calculated.	
SiO_2	46.62	53.71	53.86	54.06
Al_2O_3	26.04	29.94	30.52	30.43
CaO	1.48	0.34	—	—
K_2O	none	—	—	—
Na_2O	15.67	0.37	—	—
NH_3	—	9.26	9.85	10.14
H_2O at 100° ..	0.39	0.42	—	—
H_2O above 100° ..	10.18	5.94	5.77	5.37
	100.38	99.98	100.00	100.00

It may not be superfluous to note that the water given in the last two columns represents the difference between ammonia and the hypothetical ammonium oxide which has replaced soda.

Two other experiments upon natrolite remain to be noticed. First, the fresh mineral was boiled for fifteen minutes with a 25 per cent sodium carbonate solution; 0.72 per cent of silica dissolved. Similar treatment of ignited natrolite took out 0.62 per cent. No silica is split off by ignition. Ammonium-natrolite, before ignition, yielded 0.85 per cent of soluble silica, and after ignition 0.86 per cent. Here again no silica had been split off from the molecule, and practically none was liberated by the action of the ammonium chloride upon the natrolite. A simple, direct substitution of ammonium for sodium had occurred.

Scolecite.

On account of the well recognised analogy between natrolite and scolecite, the latter mineral seemed to be peculiarly worthy of examination. The specimen at our disposal was a mass of stout, radiating needles, which was collected by one of us at Whale Cove, on the island of Grand Manan, New Brunswick. Scolecite, we believe, has not hitherto been recorded from this locality, and on this account alone the material deserved attention.

Three sealed tube experiments were carried out, essentially as in the case of natrolite, as follows:—

- Heated ten hours at 350° . 13.74 per cent of lime and 0.35 of soda were taken out. The residue contained 8.78 per cent of ammonia.
- Heated ten hours at 370° . 12.97 of lime and 0.22 of soda were extracted. 8.48 per cent of ammonia in the residue. On account of the excessive temperature of this experiment, some reversion of the converted material had taken place.
- Heated five hours at 340° – 350° . Leach not studied. 8.91 per cent of ammonia in residue.

Analyses of the scolecite and of residues B and C are given below. The less perfect transformation in the case of B is evident.

	Scolecite.	Residue B.	Residue C.
SiO_2	45.86	53.39	53.69
Al_2O_3	25.78	30.51	30.50
CaO	13.92	0.62	0.42
Na_2O	0.41	undet.	0.29
NH_3	—	8.48	8.91
H_2O at 100° ..	0.40	0.74	0.12
H_2O above 100° ..	13.65	6.28	6.52
	100.02	100.02	100.45

The product of the reaction is plainly the same as that obtained from natrolite, and the identity in type of the

two species is perfectly clear. This fact is further emphasised by an experiment upon the solubility of silica. The fresh scolecite gave up 0.36 per cent of silica to sodium carbonate solution, and the ignited mineral yielded only 0.50 per cent. Again, natrolite and scolecite behave in the same way.

Upon both minerals fractional determinations of the water were made, and the amount lost at each temperature was noted. The results, expressed in percentages of the original minerals, were as follows:—

Temperature.	Water lost.	
	Natrolite.	Scolecite.
100°	0.39	0.40
180°	0.40	0.52
250°	0.37	4.76
350°	8.51	0.55
Incipient redness ..	0.72	7.72
Full redness	0.12	0.04
Over blast	0.06	0.06
	10.57	14.05

Scolecite contains one more molecule of water than natrolite, and that amount, one-third of its total, seems to go off at a lower temperature than the other two molecules. Otherwise the two series of experiments are probably not far apart, and they indicate that the water is in neither case constitutional. The same conclusion is suggested by the existence of the anhydrous ammonium compound, the three formulae being as follows:—

Scolecite	$\text{CaAl}_2\text{Si}_2\text{O}_{10} \cdot 3\text{H}_2\text{O}$
Natrolite	$\text{Na}_2\text{Al}_2\text{Si}_2\text{O}_{10} \cdot 2\text{H}_2\text{O}$
Ammonium natrolite ..	$(\text{NH}_4)_2\text{Al}_2\text{Si}_2\text{O}_{10}$

The parallelism is complete, and all three compounds are evidently salts of an acid $\text{H}_2\text{Si}_2\text{O}_7$, which is probably orthotrisilic acid, $\text{Si}_2\text{O}_7(\text{OH})_2$. The second anhydride of this acid, $\text{H}_4\text{Si}_2\text{O}_7$, would be the ordinary trisilic acid of orthoclase and albite, a relation which is certainly suggestive. We do not, however, care to enter upon the question of chemical structure in this paper, and we therefore leave the subject for fuller consideration at some future time. It is clear, however, that orthosilicate formulae for natrolite and scolecite must be discarded.

Prehnite.

The prehnite taken for examination was an old specimen from Paterson, New Jersey. The analysis, with fractional water determinations, is given below.

Analysis.		Fractional water.	
SiO_2	42.31	At 100°	0.21
Al_2O_3	19.95	At 180°	0.18
Fe_2O_3	6.20	At 250°	0.10
FeO	none	At 350°	0.11
CaO	26.63	Incipient red heat ..	0.28
H_2O	5.02	Full red heat	4.05
	100.11	Over blast	0.03
		Total	5.02

With sodium carbonate solution, 0.38 per cent of silica was extracted from the fresh mineral. From the ignited prehnite 1.22 per cent was taken out. Very little silica, therefore, is liberated by ignition.

Two determinations were made of the action of ammonium chloride, as follows:—

- Heated eight hours. On leaching with water, 1.31 per cent of lime and 0.17 of alumina dissolved.
- Heated twelve hours. 1.41 per cent of lime was extracted, and in the washed residue 0.22 per cent of ammonia was found.

Prehnite, therefore, differs widely from natrolite and scolecite in its behaviour with ammonium chloride. Very little action takes place, even upon long heating to

350° in a sealed tube, and practically no ammonia is absorbed. The water is more firmly held than was the case with the other two minerals, and is almost certainly to be regarded as constitutional. The orthosilicate formula for prehnite is unaffected by these results, and may stand as fairly probable. Prehnite cannot be correlated with natrolite and scolecite on any basis of similar chemical structure.

Pectolite.

In the first paper of this series (*Amer. Journ. of Science*, October, 1899) we described a number of experiments upon pectolite, in which we showed that it was almost undoubtedly a metasilicate; but the action of ammonium chloride upon it was neglected, as having been studied already by Schneider and Clarke (Bulletin No. 113, "U.S. Geological Survey," p. 34). In their experiments upon pectolite from Bergen Hill, which was nearly identical in composition with our sample, a triple heating in an open crucible with ammonium chloride removed 20.50 per cent of lime, 6.95 of soda, and 0.54 of manganese oxide; or about two-thirds of the total amount of bases present. In our last paper we reported a preliminary experiment by the sealed tube method, and found that 20.72 per cent of lime and 6.46 of soda were taken out, while 1.44 of ammonia was retained by the residue. Here again two-thirds, approximately, of the bases had been converted into chlorides by the reaction. The open crucible and the sealed tube gave essentially the same results, although the retention of ammonia was not noticed by Schneider and Clarke.

In order to obtain further light upon pectolite we continued our experiments with the sealed tube method, and have obtained very variable results. All of the heatings with ammonium chloride were conducted at 350°, and the pectolite used was from the same Bergen Hill specimen which served us for our previous work. Our data are as follows, including, for convenience of comparison, the preliminary experiment which was cited above.

- Heated six hours. On leaching, 20.72 per cent of lime, 6.46 soda, and 0.11 alumina dissolved. The residue contained 1.44 per cent of ammonia.
- Heated six hours. 20.10 per cent lime and 5.80 of soda extracted. 1.45 per cent ammonia in the residue. The residue was also examined for silica soluble in 25 per cent sodium carbonate solution (on fifteen minutes boiling), and 43.38 per cent was found.
- Heated six hours. Soluble portion neglected. The residue contained 2.23 per cent of ammonia and 61.79 per cent of soluble silica. The full analysis of this residue is given later.
- Heated ten hours. A complex breaking up of the pectolite took place, and leaching with water extracted the following percentages:—

SiO ₂ ..	..	..	..	5.43
Al ₂ O ₃	..	..	..	0.22
CaO ..	..	..	..	28.20
MnO ..	..	..	..	0.23
Na ₂ O ..	..	..	..	8.28

The residue from this leaching contained 39.63 of soluble silica, but ammonia was not determined.

These results are so irregular that definite conclusions can hardly be drawn from them. A and B agree fairly with each other, and also with the earlier work of Schneider and Clarke. C contains more ammonia, but differs widely from B as to the amount of soluble silica in the residue. D, which represents a long heating, indicates a more complete reaction than was observed in either of the other cases.

An ammonium compound, however, is evidently formed during the reaction, although its precise nature cannot be determined from the evidence now in hand. Something may be inferred from the following figures, which are to be summarised thus:—First, we reproduce from our earlier

paper the analysis of the pectolite itself. Secondly, we give the analysis of the insoluble residue obtained in experiment C. The third column of figures is obtained by subtracting from the second column 61.79 of soluble silica and 1.18 of hygroscopic water, and re-calculating the remainder to 100 per cent. The fourth column contains the molecular ratios calculated from the third.

	Pectolite.	Residue found.	Residue reduced.	Ratios.
SiO ₂	53.34	75.98	37.74	0.629
Al ₂ O ₃	0.33	0.08	0.19	0.002
CaO	33.23	9.56	25.43	0.454
MnO	0.45	0.24	0.63	0.009
Na ₂ O	9.11	1.84	4.89	0.079
NH ₃	—	2.23	5.93	0.349
H ₂ O at 100° ..	0.27	1.18	—	—
H ₂ O above 100° ..	2.70	9.47	25.19	1.399
CO ₂	0.67	—	—	—
	100.10	100.58	100.00	

These ratios roughly suggest the formation of a salt approximating in composition to the formula—



in which R' is about two-thirds ammonium and one-third sodium. Pectolite itself has the formula $NaHCa_2Si_3O_9$; so that the existence of a hydrous ammonium pectolite is indicated—a conclusion which is probable, but not proved. The reaction between pectolite and ammonium chloride is possibly simple at first, but followed by or entangled with secondary changes which obscure the results. The experiments are interesting, however, as showing how widely pectolite differs from the other minerals which we have studied, as regards the ammonium chloride reaction. The general investigation is to be continued, and we hope to take up next the more important lime-soda zeolites.

NOTICES OF BOOKS.

Quantitative Chemical Analysis. By Dr. C. REMIGIUS FRESSENIUS, Privy Aulic Counsellor, Director of the Chemical Laboratory at Wiesbaden, Professor of Chemistry, Physics, and Technology at the Wiesbaden Agricultural Institute. Seventh Edition. Translated from the revised Sixth Edition by CHARLES E. GROVES, F.R.S. Vol. II. London: J. and A. Churchill, 7, Great Marlborough Street. 1900.

We are glad to announce that this valuable work is now complete. The publication has occupied some considerable time, but the fact that it has appeared in seven parts will partly account for this. The book is too well known to need much comment; no effort seems to have been spared to bring the work thoroughly up to date, and we have already referred to the various parts as they have been published. From Part II. it is not merely a new edition, but is in reality a new book. The "special part" includes the analysis of potable waters, mineral waters, technical products, and minerals, as well as the estimation of sugars, alcohol, tanning materials, and anthracene, the estimation of the inorganic matter in plants, the analysis of soils and manures, and the examination of atmospheric air. The revision of the second part afforded the late author of the book an opportunity of introducing the various analytical processes which had been published since the appearance of the former edition. Although the results of the re-determination of atomic weights have been embodied in the tables given at the end of the book we notice that the decomposition of didymium has been overlooked. This is a pity, as the determination by Brauner and others of the equivalents of its constituents

neo- and praseo-dymium have placed the matter quite beyond doubt. We note that lanthanum is called lanthanum, but this is probably a clerical error.

The exercises for practice at the end of the book, and also the tables for the calculation of analyses will be found of very great value. The book is plentifully and well illustrated, and there is a full table of contents and a copious index that will greatly facilitate its use.

CORRESPONDENCE.

XANTHATES OF NICKEL AND COBALT.

To the Editor of the Chemical News.

SIR,—Some chemists in the United States are, apparently, claiming to have discovered, in 1895, the easy method of separating nickel from cobalt by means of xanthate of potash, which method was discovered by me in 1877, and duly published in the *CHEMICAL NEWS* of that year.

Since the publication of my note ("Observations on some Xanthates: Separation of Nickel and Cobalt," 1877) I have replied to private correspondence, and given instructions on the subject to several chemists at home and abroad.—I am, &c.,

T. L. PHIPSON, Ph.D.

Casa Mia Laboratory, Putney, S.W.,
August 18, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

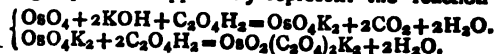
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxi., No. 4, July 23, 1900.

Solubility of a Mixture of Salts having a Common Ion.—Charles Touren.—The author considers the two cases:—(1) Of the solubility of solid potassium nitrate in solutions of increasing concentration of potassium carbonate, and (2) of the solubility of potassium nitrate in solutions of increasing concentration of potassium bicarbonate. In the first case, the numbers prove that the dissociation of the nitrate and of the carbonate is not complete; the chloride and the carbonate only lower the solubility of the nitrate for mean concentrations. In the latter case, where the uncertainty of the nature and the number of the ions is greater than in the preceding case, the curve of solubility obtained appears to be decidedly above the curve relative to the chloride.

A New Complex Acid and its Salts. Pallado-oxalic Acid and Pallado-oxalates.—H. Loiseleur.—This paper describes four new bodies: pallado-oxalic acid and three of its salts—silver, sodium, and barium pallado-oxalates. The preparation of pallado-oxalic acid is worthy of attention, as it is the only complex acid of palladium discovered up to the present time. Since the researches of Roessler, who vainly tried to isolate pallado-hydrocyanic acid, palladium was reputed incapable of forming complex acids and as a conclusion from this, appeared to possess only in a very slight degree the metalloidal character which platinum shows decidedly in so many of its combinations. The present research shows that this is not true. It is even apparent that this metalloidal character is more marked in palladium than in platinum, since pallado-oxalic acid can be obtained in very well-formed crystals, whilst the corresponding platinum, plato-oxalic acid, has only been obtained by Söderbaum in the form of a confused crystalline mass.

Some Osmyloxalates.—L. Wintrebert.—When excess of oxalic acid acts on a potash solution of OsO_4 , a new salt is formed which acts on polarised light. During the course of this reaction, when large quantities of CO_2 are evolved, a violet colouration is apparent, which is characteristic of potassium osmate, OsO_4K_2 . By adding oxalic acid to an alkaline concentrated solution of potassium osmate, the liquid, when slightly heated, passes from dark red to clear brownish yellow, and on cooling deposits brown needle-shaped crystals. Analysis shows these crystals to have the formula $\text{OsO}_2(\text{C}_2\text{O}_4)_2\text{K}_2 \cdot 2\text{H}_2\text{O}$, and is a hydrated osmyl-oxalate of potassium. The following equations apparently represent the reaction:—



An osmyloxalate of sodium was also prepared.

Action of various Finely-divided Metals (Platinum, Cobalt, and Iron) on Acetylene and Ethylene.—Paul Sabatier and J. B. Senderens.—In a previous paper the authors showed that recently reduced nickel acts directly on acetylene, either slowly or with incandescence. With platinum black, iron, or cobalt the slow reaction is scarcely perceptible, and the phenomenon is limited to the decomposition with incandescence followed by the more or less complete hydrogenation of a portion of the acetylene. Ethylene, on the other hand, is rapidly decomposed above 300° by recently reduced nickel. The metal swells up considerably and evolves a variable mixture of methane, ethane, and hydrogen accompanied by a very slight proportion of higher formenic carbides.

Synthesis of Paramethoxyhydratropic Acid.—J. Bougault.—This acid has been synthesised already by M. Trinius, but owing to some errors the synthesis has been again undertaken by the author, who arrives at the following conclusions:—(1) M. Trinius made an error in identifying with phloretic acid the paraoxyhydratropic acid which he had obtained synthetically, starting from atropic acid. (2) This paraoxyhydratropic acid is identical with that which results from the demethylation of the acid which the author prepared when starting from anethol. (3) The derived acid of anethol is paramethoxyhydratropic acid.

Influence of Hydrobromic Acid on the Velocity of the Reaction of Bromine on Trimethylene.—G. Gustavson.—Hydrobromic acid possesses the remarkable property of exciting the reaction between trimethylene and bromine. The author examines this reaction quantitatively.

Organic Solutions of Iron Perchloride.—E. Schaner de Coninck.—When organic solutions of Fe_2Cl_6 are filtered through animal charcoal under certain conditions, it is observed that the water, even when present in small proportion, exercises a decomposing action which is brought into play by the animal charcoal. Methyl alcohol acts in the same way, and is in a manner more efficacious than when it is present in larger quantities. Organic liquids, which do not contain either water or methyl alcohol, simply dissolve the iron salt, and a very long time is necessary to produce even a slight decomposition of the chloride.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxi., No. 7.

This number contains no fresh matter of chemical interest.

No. 8.

Preparation of Fluorine by Electrolysis in a Copper Vessel.—H. Moissan.—Already inserted in full.

Production of Ozone by the Decomposition of Water by means of Fluorine.—H. Moissan.—Already inserted in full.

Action of Hydrofluoric Acid and of Fluorine on Glass.—H. Moissan.—Already noticed.

Volume Composition of Hydrofluoric Acid.—H. Moissan.—Already noticed.

The Constitution of Isolauroic Acid.—G. Blanc.—Already noticed.

An Acid Dihydrodisulphone Derivative of Carvone.—H. Labbé.—It has been supposed up to the present that carvone was not susceptible of forming a combination with the alkaline bisulphites. Recent examples have, however, shown, in the aldehydic series, that the non-saturated bodies of this class possess the power of fixing, besides the normal molecule of bisulphite, one or two other molecules which become attached to the double bonds of the substance. The author has shown that certain cyclic ketones give relatively very stable compounds, which are not decomposed by acids or alkalis. Carvone is one of these, and it is easy to prepare a dihydrodisulphone derivative from this ketone. The carvone is treated at the boiling temperature in a flask fitted with a vertical condenser with a solution of commercial bisulphite of soda with enough dry carbonate of soda added to remove the sulphurous acid. At the end of three-quarters of an hour the ketone is entirely dissolved. To isolate this dihydrodisulphone derivative, the aqueous solution is evaporated almost to dryness on the water-bath, taken up with alcohol, boiled again, and filtered rapidly in a warm funnel. The evaporated alcoholic solution is again taken up with alcohol at 97–98°, filtered, and evaporated a third time; taken up again with alcohol as before, and finally evaporated quickly to dryness in vacuo. The dihydrodisulphonate of sodium carvone is thus obtained in the form of a white or yellowish white powder, which is very deliquescent; its composition is found on analysis to be $C_{10}H_{16}O_7S_2Na_2$.

Rapid Method for the Estimation of Carbonic Acid in different Gases.—L. Vignon and L. Meunier.—Already noticed.

An Apparatus for Measuring Disengagements of Gas at a Constant Volume.—A. Job.—This new gasometric apparatus is based on the following principle:—A closed vessel of constant volume is furnished with a pressure gauge. If, in the interior of this closed space, we produce a reaction which gives off a gas, and we bring the apparatus back to the original temperature, the excess of pressure, reduced to 0°, will be proportional to the mass of the gas given off. A special arrangement enables us to bring bodies with which we wish to react in contact with the gas by means of an exterior manipulation which does not alter the gaseous mass contained in the apparatus. A description of the apparatus follows, which requires the accompanying figure to be properly understood. By means of this apparatus estimations of carbonates can be made with great exactitude, and, from a practical point of view, it has an important application in the estimation of ammoniacal salts, and of urea by the disengagement of nitrogen. As a ureometer, for which purpose a special pattern has been designed, it has already been of great service.

New Electrodes for Electrolytic Estimations.—A. Hollard.—This apparatus cannot be properly described without the accompanying diagram; it has, however, the advantage of giving a deposit of regular thickness on both the interior and exterior of the cone-shaped electrode, with an increase of speed in the formation of the deposit. The gas is given off throughout the mass of the liquid, and thus losses by projection are less to be feared, and at the same time a much stronger current can be used than is generally the case.

Analysis of Commercial Copper.—A. Hollard.—Already inserted in full.

The Localisation, Elimination, and Origin of Arsenic in Animal Tissues.—A. Gautier.—Already noticed.

Estimation of Arsenic in Metals and in Alloys.—A. Hollard.—Already inserted in full.

Reversible Liquefaction: a New Physico-chemical Property of Albumenoid Substances.—M. Tavett.—Already noticed.

Journal de Pharmacie et de Chimie,
Series 6, Vol. xi., No. 12.

Santalenes and Santalols.—M. Guerbet.—When heated to 180–190° in sealed tubes with acetic acid (fusing-point 16°), the α and β santalenes combine very slowly with the acid, giving acetates of santalene. After heating for 120 hours only 1.25 grms. of acetate of santalene α is obtained from 50 grms. of the carbide and 20 grms. of acetic acid. If a current of dry hydrochloric acid gas is passed through an ethereal solution of the santalene, cooled with a mixture of ice and salt, two liquid hydrochlorates are obtained answering to the formula $C_{15}H_{24}2HCl$. These hydrochlorates cannot be distilled, even in vacuo, without decomposing. Both the α and β santalenes combine with the chlorides of nitrosyl, giving nitroschlorides with the formula $C_{15}H_{24}NOCl$. The different properties of the santalenes show that they are sesquiterpenes. The α and β santalols are both primary alcohols, their speeds of etherification are 41.7 for the isomer α and 43.6 for the isomer β , their limits of etherification are 68.5 for the α santanol and 69 for the β santanol. This fact is interesting, as all the terpenic or sesquiterpenic alcohols known up to the present have been regarded as secondary or tertiary alcohols.

MISCELLANEOUS.

Reduction of Tungstic Anhydride by Zinc. Preparation of Pure Tungsten.—Marcel Delépine.—The reduction of tungstic anhydride by means of zinc allows of the easy preparation of pure tungsten at temperatures below that at which zinc distils. In physical properties, the metal thus prepared possesses the density and heat of combustion of crystalline or ordinary tungsten. It can also, by compression or trituration, be made to assume the brilliance of metals. In fact, it can be positively asserted that this substance acts as an identical element, though prepared in a state of fine division due to the slight fusibility of tungsten.—*Comptes Rendus*, cxxxi., No. 3.

The Use of Alkaline Chlorides in the Absorption of Phosphide of Hydrogen, and on a Method for the Purification of Raw Acetylene based on this use.—C. Goettig.—The purification of raw acetylene is generally carried out by passing the gas through saline solutions which absorb the PH_3 , NH_3 , SiH_4 , CO , &c. In such cases the formation of a detonating metallic acetylide is to be feared; therefore these solutions, which are, further, easily exhausted, are made strongly acid. The author, by adding an alkaline chloride to these solutions, increases the time of their use, and, moreover, permits the use of neutral solutions. For example, a solution of ferric nitrate, sulphate of copper, or mercuric nitrate, acidulated with nitric acid, becomes cloudy after passing about 600 c.c. of raw acetylene. After the addition of about 40 per cent of chloride of potassium, it takes 2600 c.c. of the gas to make the solution cloudy. A non-acidulated solution of mercuric chloride becomes cloudy after passing 1200 c.c. of the raw gas; after the addition of KCl it takes nearly 6000 c.c. of the gas to produce a cloudiness. We see that the use of KCl has prevented the formation of a metallic compound for a considerable time without the use of nitric acid.—*Berichte*, vol. xxxii., p. 1879.

Preparation of Antitoxin from Antidiphtheritic Serum.—E. Freund and C. Sternberg.—After having shown, in this note, the action of a large number of alkaline salts, or of salts of the heavy metals, on antidiphtheritic serum, from the point of view of the precipitation and the destruction of the antitoxin, the authors propose the following method for the preparation of this antitoxin. The serum is treated with one-third its volume of a solution of potash-alum at 5 per cent; the precipitate formed is collected by filtration; the alum is removed from the filtrate by dialysis, which is then thrown on a filter to remove the slight precipitate formed during the dialysis; it is then treated with an equal volume of a saturated solution of sulphate of ammonia. The precipitate formed is re-dissolved, submitted to prolonged dialysis, and concentrated in vacuo. In this manner we can obtain, from half a litre of serum, about 9 grms. of a dry, brownish, gelatinous residue, possessing antitoxic properties, completely soluble in water or in physiological salt water.—*Zeit. f. Hygiene*, vol. xxxi., p. 429.

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NOTICE.

The **STUDENTS' NUMBER** of the **CHEMICAL NEWS** will be published on Friday, September 21st. Gentlemen holding official positions in the Universities, Medical Schools, &c., of the United Kingdom, where Chemistry and Physical Science form a part of the education, who have not yet forwarded the necessary information to our Office for publication in that Number, will confer a favour by sending it with the least possible delay.

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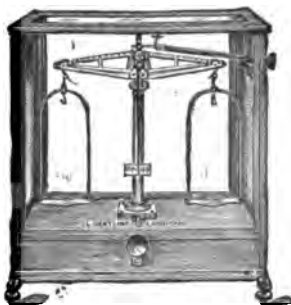
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2127.

ON THE ANALYSIS OF CHROME-IRON ORE BY THE BORAX METHOD.

By R. W. EMERSON MACIVOR, F.I.C., &c.,
Formerly Assistant-Professor of Chemistry, Anderson's College,
Glasgow.

OVER a quarter of a century ago the late Professor Dittmar and myself had occasion to make complete analyses of numerous samples of chromite, and at first employed the acid sulphate of potassium process to effect the decomposition of the ore, but soon abandoned it in favour of a modification of the then generally neglected borax process. In later years, long after my professorial connection with him had ceased, Dittmar further improved this process, and it is a matter of some surprise to me that the details of his method should be absent from the pages of our principal laboratory text-books. The process is not only an eminently practical one for commercial purposes, particularly when complete analysis of the ore is required, but, in my opinion, forms a good quantitative exercise for the analytical student.

I am prompted to send this note for publication in the CHEMICAL NEWS through having read R. Fieber's recent paper on "A Simple Method of Decomposing Chromite" (*Chem. Zeitung*, xxxiv., 333, and *Amer. Journ. Sci.*, 1900, vol. x., No. 55), in which the borax process is recommended for the purpose indicated in the title of the paper.

Fieber's manner of fluxing his chromite is briefly as follows:—0.5 gm. of the finely powdered ore is fused in a platinum crucible with 3 grms. of sodium-potassium carbonate, for ten minutes; when cold, 3 grms. of dry borax are introduced, and the whole is again fused by first heating for a short time over the flame of a Teclu burner, and then for forty-five minutes over the blowpipe. If the decomposition is not then complete, a further quantity of the mixed carbonates should be added and the operation of fusing repeated.

I fail to see any material difference between Fieber's method and that described in text-books which I may now call ancient, and, on his own showing, it possesses the same defects, the chief of which is uncertainty as to the number of fusions necessary to effect the decomposition of the ore. Now in Dittmar's improved borax process there is no doubt on this score, provided, of course, the ore is rendered impalpable and the manipulations carried out in accordance with his directions. The experience of many years, with chrome ores from all parts of the world, enables me to confirm—if confirmation be at all necessary—Dittmar's own statement: "Undecomposed ore rarely exceeds 2 m.grms. (per gm. of ore = 0.2 per cent) if the fusion be carefully conducted. Such small quantities may be taken into account as containing 50 to 60 per cent Cr_2O_3 without serious error."

The main feature of the Dittmar process is the use of a flux composed of equal weights of borax glass and the carbonates of potassium and sodium (the latter mixed in the proportions of their molecular weights) prepared beforehand by melting the components together in a platinum dish or crucible, and pouring out the melt on a suitable polished surface. The flux is broken up into pieces, and, being somewhat hygroscopic, should be kept in well-stoppered bottles. I have at times varied the composition of this flux, but the relative proportions have never been greater than 3 parts of KNaCO_3 to 2 parts of borax. This mixture gives a flux of more "pasty" consistency,

and therefore better able to keep the ore in suspension during fusion, thereby facilitating its decomposition.

It would not, of course, be permissible in a contribution like the present to enter into the full details of Dittmar's process for the complete analysis of chromite, but as few, if any, of our analytical works refer to it at all, perhaps the particulars of its first stage may be of interest to some of the readers of this journal:—Fuse 4 grms. of the flux in a platinum crucible over an Iserlohn lamp, allow to solidify, and put on the top of the fuse about 0.5 gm. of the ore, which must be in an impalpable condition. Heat the crucible with the lid on until the flux has quite melted and the ore sunk to the bottom. The lid is then taken off, the crucible placed at an angle on a triangle, and the lid in front of it horizontally; heat is again applied, and the contents of the crucible kept in motion by aid of a thick platinum wire until the ore is dissolved. If the lamp is in good order and the gas pressure high, it will not be necessary to have recourse to the blowpipe. After half an hour's heating the whole of the Cr_2O_3 can be assumed to have passed into the state of alkaline chromate. The contents of the crucible are allowed to cool to solidification, 2.5 grms. Na_2CO_3 added, and the whole again fused. The fuse is poured into a platinum dish, and the crucible freed from adhering matter by heating in water contained in a porcelain basin, and thorough washing. To this solution the bulk of the fused mass is added, and the whole allowed to stand on a water-bath until disintegration is complete; filter, wash with hot water as long as the runnings are yellow.

When a complete analysis is contemplated it is well to start with a gm. of ore. This involves the use of twice the quantities of flux and Na_2CO_3 recommended for 0.5 gm. of ore.

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GADOLINIUM.

By EUG. DEMARCAV.

GADOLINIUM, discovered by Marignac, and which M. Lecoq de Boisbaudran characterised by its beautiful band spectrum (*Comptes Rendus*, cxi., p. 472), has since been the object of researches by Bettendorf (*Liebigs Annalen*, cclxxx., p. 376), and recently by Benedick (*Zeit. f. Anorg. Chem.*, xxi., p. 393).

The gadolinium used in these researches contained manifestly, besides small quantities of terbia, shown by the yellow colour of the oxide (which is white in the pure state), a certain quantity of $\Sigma\text{-Zr}$, which they have not eliminated.

During the course of my fractionations I isolated a decided quantity of double nitrate of magnesium and gadolinium. The spectrum of the gadolinium extracted from this salt, examined under the spark of my induction coil, only showed slight traces of the strong lines of $\Sigma\text{-Zr}$ and yttrium. The stronger lines of this spectrum are only feeble; I estimated these traces of yttrium at less than 1/10,000th part. In the absorption spectrum, absolutely nothing is visible between 3500 and the red end when a layer of 15 cm. thick of melted nitrate is used, and the same on reversion.

This gadolinium at first sight appears to be as white as magnesia, but with a little attention slight yellow tinges may be seen. This faint tint probably shows the presence of a trace of terbia, for these two earths are very difficult to separate; the other salts are colourless. The double magnesium nitrate melts at 77.5–78°. The simple nitrate, crystallising with 6H₂O, melts about 91.5–92°. This last number is a little uncertain; at this temperature the nitrate with 6H₂O seems to lose water and be transformed into a less hydrated nitrate, which remains in solution and may lower the fusing-point.

This gadolinium gives the band spectrum of M. Lecoq de Boisbaudran. This method of spectrum examination is, however, not so reliable for this research as the photographic method of the line spectrum. Between the limits $\lambda=4800$ and $\lambda=3500$ the principal lines of this spectrum, which have not been described before, are as follows:—

λ .	Strength.	λ .	Strength.
4583.1	7 (a)	3867.3	7
4522.5	7	3863.2	6
4346.8	10	3852.6	11
4342.3	9	3844.8	9
4316.5	6	3842.5	9.5
4296.8	10	3840.0	8
4281.1	9	3837.3	7
4262.9	11	3835.2	7
4252.7	8	3827.6	6
4239.8	7	3817.0	6
4230.9	6	3814.2	10
4218.4	7	3805.6	9
4216.2	7	3796.8	11
4213.0	8	3792.5	8
4205.8	7.5 (b)	3791.5	8
4198.6	8	3788.1	8
4191.8	8	3782.7	9
4184.8	10	3771.2	9
4176.2	6	3768.9	11
4163.3	6.5	3764.8	7
4155.3	6	3761.3	6
4140.9	6	3744.0	10
4137.3	8	3740.6	7
4132.4	10	3733.6	7
4130.3	11 (c)	3731.4	7
4111.6	7	3719.9	9
4098.6	13	3713.1	8
4093.8	6	3698.1	7
4087.7	6.5	3671.4	7
4085.7	9	3664.7	10
4078.3	10	3662.4	6
4073.7	10	3656.3	8
4070.4	8	3654.7	8
4063.4	9	3651.1	6
4058.2	6	3646.3	11
4053.7	9	3634.6	6
4049.8	12	3624.9	6
4039.7	8	3617.1	6
4037.6	9	3613.4	6
4028.2	7	3610.9	7
4023.4	6	3608.8	6.5
4013.9	7	3600.9	6
4009.1	8	3592.6	7
4005.2	7.5	3584.9	9
4001.6	6	3581.8	7
3994.4	6	3578.3	6
3987.9	6.5 (d)	3574.7	6
3974.7	6	3571.8	6
3959.9	10	3558.2	7
3958.0	9.5	3557.0	7
3916.7	10	3549.3	10.5
3902.6	8	3545.7	10
3886.1	9	3542.6	6
3895.0	9	3511.7	9

(a). Near to a strong line of $\Sigma-Z_4$.

(b). Coincides with a strong line of $\Sigma-Z_4$.

(c). Coincides with a strong line of $\Sigma-Z_4$.

(d). Very near to a strong double line of ytterbium.

The maximum of strength for this scale is 16 and the minimum 1.

In this spectrum the most persistent lines are 3549.3 and 3545.7, and these, with the other strong lines of the spectrum, constitute a good reaction for gadolinium. This reaction, although very inferior in sensibility to that for yttrium, ytterbium, and $\Sigma-Z_4$, allows of the detection of gadolinium directly in almost all crude rare earths.

The atomic weight of gadolinium has been found to be about 155 by the various chemists who have investigated the properties of this element. From several attempts I have also found a number approximately the same. I believe it to be slightly too high, in consequence of the defects of the method (synthesis or analysis of the anhydrous sulphate) used by myself and all the other observers.—*Comptes Rendus*, cxxxi., No. 5.

ATOMIC WEIGHT OF RADIO-ACTIVE BARIUM.

By M^{ME}. CURIE.

THROUGHOUT our researches on the isolation of radium, the progress of the concentration of this element has been controlled by examination of the spectrum, and by determinations of atomic weights.

Each time that a quantity of radiferous barium chloride was obtained from the mineral, I submitted this chloride to a systematic fractional crystallisation to extract several decigrams of a product containing more concentrated radium. By adding to the solution of this product a little alcohol, or better, a little hydrochloric acid, until a slight precipitate forms, I obtained one or two centigrams of a still more concentrated product, which was used for the spectrum examination. The remaining product served for the determination of the atomic weight of radiferous barium.

The investigation of the spectrum of the successive products has been undertaken by M. Demarcay, who has published his results. The last of these products apparently only contains a trace of barium, and may be considered as almost pure radium chloride.

The isolation of radium chloride has therefore been obtained, but the quantity of pure salt isolated is insufficient for the determination of the atomic weight of radium. I had to be content with determining the atomic weight of radiferous barium with the less concentrated product, of which I had 0.4 grm.

The last analogous determination, which I made several months ago, gave for the atomic weight of radio-active barium the number 146, which already is decidedly higher than 137.5, the atomic weight of pure barium.

The method now employed was the same. It consists in the estimation of the chlorine in the anhydrous chloride. A determination was simultaneously made on pure crystalline barium chloride under exactly the same conditions, this latter serving as a control experiment. The pure barium chloride was weighed in a crystalline state, also in the anhydrous state; the radio-active barium chloride only in the anhydrous state.

After being kept at a temperature of 130° for an hour, the two salts lose all their water of crystallisation; after this their weight remains constant, however long the heating is continued. Even if the temperature is raised to 150°, and kept so for an hour or more, no further loss of weight is observed, and this indicates that there is no loss of chlorine.

This last determination gives 138.0 as the atomic weight for barium in pure barium chloride, whilst the two determinations carried out on the radio-active chloride give 174.1 and 173.6 as the numbers for the atomic weight of the metal in this chloride. We have no means of distinguishing the relative quantities of radium and barium in this product. M. Demarcay thinks, however, from the aspect of the spectrum, that there is rather more radium than barium. In any case it is certain that the atomic weight of radium is much higher than 174.

The quantity of radium chloride that I have isolated is insufficient to make an examination of the properties of pure radium. However, M. Curie and myself are very pleased at having obtained a proof of the existence of this

element, and so having confirmed the ideas which guided us in our researches on radio-active materials.—*Comptes Rendus*, cxxxi., No. 6.

THE FOSSIL FLOUR OF SANTAFIORA.

By G. DE NEGRI.

FIFTY years ago G. Fabroni proposed the manufacture of light bricks similar to those made by the ancient Romans, and which, according to Pliny, Vitruvius, and Strabo, had the property of floating on water. The material used by M. Fabroni for making his refractory bricks was the fossil flour (Berg-mehl) of Santafiora.

The bricks, prepared by the firm of Rimbotti and Hemmeler by means of this flour, which exists in large quantities in the district of Monte Amiata, are used for lining ovens.

Although these bricks resist a very high temperature without melting, they have the drawback of not possessing sufficient strength to resist the mechanical action of the materials melted in metallurgical furnaces.

The fossil flour of Santafiora is composed as follows:—

Water and organic matter		12.40
Silica		82.00
Alumina and oxide of iron		4.20
Lime		0.55
Magnesia		0.45
Undetermined and loss		0.40

By calcining at a red heat it loses 12.40 per cent, from which must be deducted 4.70 per cent of water driven off at 100°.

The calcined fossil flour has therefore the following composition:—

Silica		93.16
Alumina and oxide of iron		4.79
Lime		0.62
Magnesia		0.51
Undetermined and loss		0.92

This fossil flour, by its state of extreme division, by its porosity, and by its richness in silicic acid, is easily attacked by the alkalis, and can be used for the manufacture of silicate of potash and silicate of soda (soluble glass).

This operation, at the ordinary pressure, is fairly long, but if alkalis of 1.2 to 1.3 density are used under a pressure of about three atmospheres for two or three hours, we obtain a soluble glass which is richer in silicic acid than that prepared by the use of silica.

The natural fossil flour is more readily attacked by alkaline solutions than that which has been calcined, but the glass obtained by means of this latter, for the same amount of alkali, contains more silica, as the raw fossil flour loses water and organic matter by calcination, and the quantity of silicic acid thus remains higher.

With natural fossil flour we obtained a soluble potash glass, which contained three parts of silica for two parts of potash. With soda we obtained a glass containing equal parts of alkali and silicic acid.

The same experiment has been repeated with fossil flour calcined at a red heat. With potash lye it gave a soluble glass which, for one part of potash, contained 1.9 of silica; that prepared with soda was formed of one part of alkali and two parts of silicic acid.

The soluble glasses obtained with this material are of excellent quality. They will bear comparison not only with those prepared by fusion, but also with the soluble glasses manufactured by Kuhlmann's process.

Fossil flour should be calcined at a high temperature, on account of its low density and its large volume. As it is a very bad conductor of heat the operation is excessively

long, and it is for this reason that the use of this material has been abandoned.

However, the new methods of calcination due to Grünt and Hagemann have considerably reduced the cost of calcined fossil flour, to such a point even as to render its use possible for the manufacture of soluble glass.

In the Monte Amiata district we also meet with another variety of fossil flour with a slight yellow colour, which is less porous and a little more compact than the other, but of a somewhat similar composition:—

Water and organic matter		12.90
Silica		80.32
Alumina and oxide of iron		5.17
Lime and magnesia		0.74
Carbonic anhydride		0.55
Undetermined and loss		0.32

It loses 7.37 per cent by calcination, after making the necessary deduction for water lost at 100°.—*Moniteur Scientifique*, Series 4, vol. xiv., July, 1900.

THE DETECTION OF NITROGEN IN ORGANIC SUBSTANCES CONTAINING SULPHUR.

By E. TAUBER.

ABOUT twenty years ago O. Jacobsen proposed a method for the detection of nitrogen in organic substances containing sulphur, consisting of adding to the substance under examination at least four or five times its volume of iron filings before heating with metallic potassium. The object of adding the iron was to transform the sulphocyanate of potassium, which was formed in the presence of nitrogen and sulphur, into cyanide of potassium and sulphide of iron.

Jacobsen's method is still considered to be good, and is still used by chemists. I was therefore astonished to find a few months ago that the addition of iron might lead to the greatest errors in the search for nitrogen in organic matters. For this reason, and also because it is entirely superfluous, the use of iron ought to be completely stopped.

At the temperature of the reaction, finely divided iron has the property of fixing the atmospheric nitrogen to such a large extent, that under the conditions laid down by Jacobsen cyanide of potassium is formed, even though the substance examined is quite free from nitrogen. I was made aware of this fact while looking for nitrogen in an organic substance containing a very small quantity of sulphur.

While in the absence of iron the substance hardly gave a faint blue colouration, the test repeated in the presence of iron furnished a voluminous precipitate of Prussian blue. It seemed very improbable that a trace of sulphur could mask such a considerable quantity of nitrogen, and the idea immediately occurred to me that the iron might have effected the fixation of the nitrogen and the formation of cyanide of potassium. Experiments fully confirmed this supposition, and showed, further, that all kinds of iron exercised the same influence from the qualitative, if not the quantitative point of view, on the formation of cyanide.

Even pure iron, free from nitrogen, obtained by the reduction of pure oxide of iron by hydrogen causes the formation of cyanide of potassium, when heated with organic substances, equally free from nitrogen. In this case the fixation of atmospheric nitrogen is beyond all possible question, while when we use impure iron containing nitrogen a portion of the cyanide formed may be due to the iron.

If, for the prevention of the access of air, we pass a current of hydrogen over the mixture containing *pure* iron during ignition, the formation of cyanide does not take

place. The proportion of cyanide formed naturally depends on the quantity of iron used, and the duration of the heating. In this manner a mixture containing very little iron, and heated for a comparatively short time, may not form any cyanide of potassium at all.

It is evident that Jacobsen had adopted this precaution—the limitation of the time of heating—without recognising its importance. It is only in this way that we can explain the fact that he did not detect the source of error caused by the use of the iron. On the other hand, the reduction to a minimum of the duration of the heating offers another danger; for the nitrogen, even when chemically combined, is not often transformed into cyanide until after an energetic and prolonged heating.

I am of the opinion that, as a rule, the formation of cyanide from nitrogenous organic matters takes place more rapidly than in the case of the atmospheric air constituting the exclusive source of the nitrogen.

From this it results that Jacobsen's method cannot give results worthy of belief, except when the mixture is heated long and vigorously in a current of pure hydrogen.

We should be all the more willing to dispense with this new complication, seeing that the use of iron for the detection of nitrogen in organic substances containing sulphur is entirely superfluous. Graebe (*Berichte*, vol. xvii., p. 1178) has already shown that the use of iron can be suppressed in this class of assay by slightly increasing the amount of metallic potassium used.

By a series of experiments I made certain that the statement made by Graebe was correct, and that when rationally employed the method always gave good results.*

If the substance contains a large proportion of sulphur, as well as a small quantity of nitrogen, the sulphide of potassium formed naturally reduces the oxide of iron added, and on the addition of acid we should obtain, in this case, not Prussian blue, but a cloudiness or bluish-white precipitate. The precipitate might consist of sulphur. To remove all doubt on this point, excess of ferric chloride should be added. The precipitate should then immediately take an intensely blue colour if ferrocyanide is present.

It is not therefore at all necessary to modify Lassaigne's method in the case of an organic substance containing sulphur. The employment of potassium in excess is useful in every case, even in the absence of sulphur.

The fact that metallic iron has the property of fixing atmospheric nitrogen, decided me to institute experiments with the object of determining whether it was not possible to base a technical method for the preparation of alkaline cyanides by means of the nitrogen of the air, on this property of iron, by substituting for the alkaline metals themselves their hydrates or carbonates, which are much less expensive. The experiments have given positive and, in a measure, such satisfactory results, that I consider the industrial use of this process not only as possible, but, further, as very probable. Certainly the results are rather variable, but I have finally succeeded in transforming with practical certainty 10 per cent of the soda used into cyanide of sodium. In some cases the return reached as much as 25 per cent.

However, in studying the bibliography of the question, I learnt that the same process had already been patented in the year 1880. The patents, belonging to Victor Adler (*German Patents*, Nos. 12351, 18945, 24334, and 32334), lapsed after six years, which shows that the process in-

volves considerable technical difficulties. In any case, basing my remarks on my own personal observations, I am still of the opinion that the process is capable of industrial application.

It may be noted in passing that no treatises or technical handbooks mention the Adler patent, while they devote a good deal of space to other much less practical attempts to utilise the atmospheric nitrogen for the manufacture of cyanides. However, it is incontestable that the atmospheric nitrogen is fixed in considerable proportions by a mixture of soda or potash and carbon in the presence of metallic iron, and that, at temperatures at which in the absence of iron the same mixtures do not give a trace of cyanide.

It is possible that, without having been aware of it, we have long utilised this particular property of iron in the manufacture of cyanide of potassium from nitrogenous scraps.

In this process we add iron to the mixture to combine with the sulphur, and thus protect the iron vessels in which the fusion takes place. But this addition of iron has probably the effect of converting into cyanide a part of the nitrogen of the scraps, which is always lost in considerable quantities during the operation.

I would further remark that among the other metals which I have tried for this purpose, no other has, to any pronounced degree, any analogous action to that of iron. I have tried copper, nickel, chromium, tungsten, and magnesium. The influence of magnesium, even in small quantities, was the most marked; then came tungsten, chromium, and nickel, and finally copper, which had no action at all at any temperature tried.

Experiments.

A. Experiments which show the Defects of Jacobsen's Method.—In all the experiments I used about 0.02 gm. of the substance and 0.2 gm. of potassium, first without iron, and then with the addition of about 0.5 gm. of iron obtained by the reduction of pure ferric oxide by hydrogen.

In some cases I made further experiments with smaller quantities of iron.

The reaction never occurred in the absence of iron, but was very pronounced in the presence of 0.5 gm. of iron after one to two minutes' heating.

The diminution of the proportion of iron had the result of diminishing the quantity of Prussian blue produced. On calcining the mixture for a very short space of time, say a quarter of a minute, the reaction does not take place, even in the presence of iron. These results were obtained with the following substances: milk sugar, sugar charcoal, hydroquinone, β naphthol, salicylic acid, phthalic anhydride, anthraquinone, and the α - and β -naphthalene-sulphonates of soda. Crystallised methylsulphonate of potassium behaved in a slightly different manner.

With 0.05 gm. of iron this salt gave no colouration, but with 0.5 gm. of iron the blue colouration obtained was very pronounced, though without being intense. The cause of this phenomenon rests, no doubt, in the fact that this substance has but little tendency to set carbon at liberty under the conditions of the experiment.

B. Experiments which show the Defects of the Lassaigne Method, even in the Presence of Sulphur.—In these experiments we also used 0.02 gm. of material and 0.2 gm. of potassium. An appreciable blue colouration, augmented by the addition of ferric chloride, was always obtained; in most cases a strong colouration is produced directly. I have examined the following bodies:—Taurine, allylsulphocarbamide, sulphocarbamide, thioparalutidine, benzidinesulphone, sulphanilic acid, naphthionate of soda, mixtures of equal parts of sulphanilic acid and sulphur, of equal parts of naphthionate of soda and sulphur, methylene blue mixed with one part of 2-amido-8-oxynaphthalene-6-monosulphonic acid and two parts of 1-oxynaphthalene-4:7-disulphonic acid, sulphonyl-sulphanilic acid, and, finally, a number of colouring

* I used about 0.02 gm. of the substance and 0.2 gm. of freshly cut potassium, and heated this carefully in a test-tube in such a manner that the metal became fused, and enveloped the substance as completely as possible. It was only then that I heated vigorously while rotating the test-tube, which is, as a rule, bent slightly out of shape under the action of the heat. To extinguish the melted mass I used 3 or 5 c.c. of water. It is because the mixture of the substance and the metallic potassium is heated too rapidly, and for too short a space of time, that mistakes in the analysis are most often made.

materials derived from triphenylmethane and azoics containing sulphur.

In none of these cases was the result doubtful, in spite of the presence of large quantities of sulphur in proportion to the nitrogen. Jacobsen's assertion to the contrary, that most of the organic substances containing sulphur as well as nitrogen will not give the Lassaigne reaction, cannot be explained by the fact that Jacobsen used too small quantities of metallic potassium. As Graebe has shown the reaction does not always succeed in these cases.

The experiments undertaken, with a view to the utilisation of finely divided iron for the fixation of the atmospheric nitrogen and the manufacture of alkaline cyanides, will form the subject of a future publication.—*Berichte*, vol. xxxi., p. 3150.

THE ESTIMATION OF ZINC AS PHOSPHATE.

By H. D. DAKIN.

THE original process for the estimation of zinc depending on the formation of zinc ammonium phosphate was first proposed by Hugo Tamm (*CHEM. NEWS*, xxiv., p. 148), and has since undergone comparatively few modifications.

Of papers published since the method was first devised perhaps the most important is that by M. Austin ("The Double Ammonium Phosphates of Beryllium, Zinc, and Cadmium in Analysis," *Amer. Journ. Sci.*, vol. viii., Sept., 1899), in which full analytical data are given. (No analyses of pure substances of known composition are given in Tamm's or in most other papers on this subject). The main conclusion drawn from the results is "that the presence of ammonium chloride is essential for the conversion of the zinc phosphate precipitated by hydrogen sodium ammonium phosphate to the ammonium zinc salt." Tolerably accurate results were only obtained when at least 10 to 30 grms. of ammonium chloride were present. The object of the present paper is to show that under somewhat different conditions this large quantity of ammonium salts is quite unnecessary; to point out, and if possible remove, certain small sources of inaccuracy; and to give test-analyses, showing that, with the prescribed conditions, good results may be obtained when weighing either as zinc ammonium phosphate or pyrophosphate.

Tamm, though strongly recommending his process, was aware of several points which he considered to be sources of error, among which may be mentioned:—

- (I.) The inclusion of alkaline salts not readily removable by washing.
- (II.) Incompleteness of precipitation.
- (III.) The impracticability of converting zinc ammonium phosphate into pyrophosphate without involving loss of zinc.

The first source of error may, as is well known, be to a large extent avoided (completely when weighing as ignited pyrophosphate) by the substitution of ammonium phosphate for the sodium salt, or possibly less completely by the use of microcosmic salt.

With regard to the second objection, that of incompleteness of precipitation, it will be seen, from the test-analyses that follow, that under suitable conditions the amount of zinc in the filtrate does not exceed two- or three-tenths of a m.grm., and is generally less.

The third objection is gone into somewhat more fully later on; but it may be mentioned here that, notwithstanding Tamm's statement, it has since been generally assumed that if care be taken during the ignition no reduction is to be feared.

For the experiments that follow, two sources of zinc to serve as a standard were used, and no difference could be detected in the results yielded by the two specimens.

The first was a purchased sample of chemically pure zinc oxide, 10 grms. of which was carefully tested for impurities with a negative result. The other specimen was obtained as follows:—Excess of pure zinc oxide was left for some days in contact with pure dilute sulphuric acid; after filtration a few drops more acid were added, and sulphuretted hydrogen passed in until a considerable precipitate of white zinc sulphide had appeared. The liquid was then filtered, and a few drops of bromine added, sufficient to produce a faint yellow colour. The solution was now fractionally precipitated with ammonia, the first portion thrown down being rejected. After filtration a portion of the remaining zinc was thrown down as basic carbonate by means of warm ammonium carbonate solution. The precipitate was well washed at the pump, dried, and ignited in a large platinum dish; then again washed with boiling distilled water, and finally ignited over the blowpipe flame in a porcelain crucible. The ignition was invariably repeated immediately before any portion of either of the two specimens was weighed out.

The ammonium phosphate employed contained slightly over 80 per cent of the di-ammonium salt, the remainder being the di hydrogen ammonium phosphate. Its solution was carefully filtered from a trace of insoluble matter. It was perfectly free from arsenic, chlorine, and other common impurities. In all cases, unless otherwise stated, the amount of phosphate employed was equal to ten times the weight of zinc known to be present.

The ammonium chloride was prepared as required by means of pure hydrochloric acid and ammonia.

In all cases precipitation was carried out in platinum vessels, in order to avoid the well-known corrosive action of alkaline phosphate solutions on glass. The bulk of liquid in which precipitation took place was about 150 c.c. The precipitate in all cases was collected on asbestos contained in a Gooch crucible, and well washed with a hot 1 per cent solution of ammonium phosphate. This was considered necessary, as it was found that prolonged contact with hot water tended to bring about partial decomposition of the precipitate, and so lead to low results. The ammonium phosphate solution adhering to the precipitate was removed by a few washings with re-distilled alcohol. The strongest alcohol should not be used for this purpose, as ammonium phosphate is insoluble in it. Cold water may, if preferred, be substituted for the alcohol, but the former has the advantage in that the precipitate dries very much more quickly.

It may here be noticed that, after filtering ammonium phosphate solutions through a filter made of the particular sample of asbestos employed, a slight, though quite perceptible, loss was observed in the weight of the filter, although the asbestos had been thoroughly well purified by treatment with concentrated hydrochloric acid; so that, when exceptional accuracy is required, it is preferable to weigh the crucible and felt after the filtration rather than before. The precipitate should be dissolved in dilute nitric acid, taking care that no fine asbestos fibres are lost, and the crucible and felt dried at 100–105° if the precipitate was weighed as zinc ammonium phosphate, or ignited and weighed in the case of the pyrophosphate. The weight of the crucible and felt, dried at 100–105° C., will invariably be found to be a few tenths of a m.grm. higher than if it had been ignited.

It is obviously of importance to be assured of the complete stability of the zinc ammonium phosphate at the temperature at which it is dried, and, as far as I am aware, no experiments have been given in support of this. The following table is therefore given. (See next column).

The numbers given represent the weight in grammes of the precipitate, dried for the length of time indicated. The numbers in brackets represent the equivalent weights of zinc.

Two specimens of the precipitate, dried for seven and ten hours respectively, both yielded 9.57 per cent of ammonia when distilled with caustic soda, the theoretical amount being 9.56 per cent. So that it may be safely

TABLE I.

Time of drying at 100—105° C.	Expt. I.	Expt. II.	Expt. III.	Expt. IV.
One hour ..	0.3509 (0.1286)	0.5174 (0.1895)	0.7454 (0.2731)	—
Two hours ..	0.3504 (0.1284)	0.5168 (0.1893)	0.7444 (0.2728)	0.3880 (0.1422)
Ten hours ..	0.3504 (0.1284)	0.5167 (0.1893)	0.7444 (0.2728)	0.3880 (0.1422)
Twenty hours	0.3504 (0.1284)	0.5167 (0.1893)	0.7440 (0.2727)	—

assumed, for all practical purposes, that the precipitate is perfectly stable at 100—105° C.

When it was necessary to ignite the precipitate, the Gooch crucible was placed inside a much larger platinum crucible, and heated at first over a very small flame, every care being taken to exclude reducing gases. The lids of the crucibles should be kept off, so as to allow of the free escape of ammonia and water-vapour, as it was found that at high temperatures the ammonia is capable of exerting a reducing action on the precipitate,—possibly due to the partial decomposition of the ammonia into its elements, which begins at 500° C. This reduction was observed by igniting portions of zinc ammonium phosphate precipitate in a porcelain boat, placed inside a porcelain tube which passed through the centre of a Fletcher furnace. At lower temperatures, such as obtain in a tube placed in an ordinary combustion furnace, little or no reduction was observable. After the ammonia and water have been to a large extent expelled, the ignition is completed by replacing the lid and heating more strongly for a short time over a full Bunsen flame. That this degree of heat is amply sufficient was proved by obtaining identical results whether the precipitate be ignited over a Bunsen or blowpipe, or in a muffle at a bright red heat. So that the use of the blowpipe, as has been advocated, is unnecessary. The pyrophosphate should be absolutely white, any greyness—sometimes observable on the surface—indicating more or less reduction.

Various methods of precipitation have been advocated at different times; they may be grouped into three classes:—

- (I.) Tamm's original method, in which a faintly acid solution of zinc ammonium chloride is precipitated by an excess of alkaline phosphate.
- (II.) The acid zinc and phosphate solutions being mixed, the whole is rendered alkaline by the addition of ammonia, the excess of which is expelled by boiling (R. C. Boyd, *School of Mines Quarterly*, xi., p. 355; also L. Jawein, *Chem. Zeit.*, xi., p. 347).
- (III.) Where the alkaline zinc and phosphate solutions are rendered neutral by the cautious addition of acid (Dr. J. Clark, *J. S. C. I.*, 1896, p. 866; also A. C. Langmuir, *Journ. Amer. Chem. Soc.*, xxi., p. 115).

It will be found that the first original method involves the least trouble, and yields results equally as accurate as those yielded by the other two modifications.

The modified process by which the following results were obtained may be described as follows:—

The acid zinc solution contained in a platinum dish is nearly, but not quite, neutralised by the addition of ammonia, diluted to about 150 c.c., and then warmed on the water-bath. (It is not advisable to heat on a hot plate or sand-bath, as, owing to the very rapidly settling nature of the precipitate, "bumping" is liable to ensue). To the warm solution ammonium phosphate solution is added, in quantity such that ten times the weight of salt is used for 1 part of zinc supposed to be present. The amorphous precipitate first produced quickly changes to a finely crystalline precipitate of zinc ammonium phosphate, the change being especially rapid in presence of considerable amounts of ammonium salts. The

heating is continued for about a quarter of an hour, and then the dish removed and allowed to stand for a short time. No difference could be detected between the results obtained when the liquid was allowed to stand for half an hour or twenty-four hours. The precipitate is next filtered off on a Gooch crucible washed with hot 1 per cent ammonium phosphate solution till perfectly free from chlorides, &c., and then washed a few times with alcohol or cold water. The precipitate is then dried at 100—105° C., and either weighed as Zn, NH₄, PO₄, or ignited, with the previously mentioned precautions, and weighed as Zn₂P₂O₇. The precipitate is then dissolved in dilute nitric acid, the crucible and felt being then weighed, after drying or igniting, under the same conditions as before.

TABLE II.

Zinc No.	ZnNH ₄ PO ₄ taken.	=Zn found.	Error.	Ammo-nium phos-phate. Grms.	Ammo-nium chlor-ide. Grms.	Zinc in filtrate.
1.	0.2856	0.7788	0.2854	-0.0002	2.9	1 trace
2.	0.2000	0.5453	0.1998	-0.0002	2.0	1 0.0003
3.	0.1219	0.3325	0.1218	-0.0001	1.2	1 trace
4.	0.1259	0.3434	0.1258	-0.0001	1.3	1 —
5.	0.2000	0.5461	0.2001	+0.0001	2.0	1 —
6.	0.1029	0.2827	0.1036	+0.0007	1.0	1 0.0001
7.	0.2598	0.7087	0.2597	-0.0001	2.6	1 0.0002
8.	0.1663	0.4531	0.1660	-0.0003	1.7	2 0.0002
9.	0.1727	0.4713	0.1727	—	1.7	2 trace
10.	0.2185	0.5946	0.2179	-0.0006	2.2	2 0.0003
11.	0.1288	0.3504	0.1284	-0.0004	1.3	2 trace
12.	0.2069	0.5653	0.2071	+0.0002	2.0	3 trace
13.	0.2826	0.7715	0.2827	+0.0001	2.8	5 0.0002
14.	0.1426	0.3880	0.1422	-0.0004	1.5	5 —
15.	0.1390	0.3781	0.1385	-0.0005	1.4	5 —
16.	0.1326	0.3603	0.1320	-0.0006	1.3	5 —
17.	0.3512	0.9585	0.3512	—	3.5	5 trace
18.	0.2044	0.5585	0.2047	+0.0003	2.0	7.5 —
19.	0.1935	0.5282	0.1935	—	2.0	10 0.0002
20.	0.1318	0.3598	0.1318	—	1.2	10 —
21.	0.1383	0.3762	0.1379	-0.0004	1.3	10 0.0001
22.	0.1509	0.4098	0.1502	-0.0007	1.5	10 —
23.	0.2000	0.5478	0.2007	+0.0007	2.0	10 —
24.	0.2163	0.5924	0.2171	+0.0008	2.1	15 —
25.	0.1877	0.5165	0.1893	+0.0016	1.8	20 0.0002

TABLE III.

Zinc No.	Zn ₂ P ₂ O ₇ taken.	=Zn found.	Error.	Ammo-nium phos-phate. Grms.	Ammo-nium chlor-ide. Grms.	Zinc in filtrate.
1.	0.1259	0.2925	0.1255	-0.0004	1.3	1 0.0001
2.	0.2000	0.4649	0.1995	-0.0005	2.0	1 trace
3.	0.1428	0.3341	0.1433	+0.0005	1.4	1 —
4.	0.1545	0.3594	0.1542	-0.0003	1.5	1 —
5.	0.2000	0.4674	0.2005	+0.0005	2.0	1 0.0003
6.	0.1029	0.2413	0.1035	+0.0006	1.0	1 0.0001
7.	0.2598	0.6045	0.2594	-0.0004	2.6	1 0.0002
8.	0.2000	0.4637	0.1990	-0.0010	2.0	1 0.0003
9.	0.1219	0.2831	0.1215	-0.0004	1.2	2 trace
10.	0.1663	0.3867	0.1659	-0.0004	1.6	2 0.0002
11.	0.1727	0.4028	0.1728	+0.0001	1.8	2 trace
12.	0.1288	0.2992	0.1284	-0.0004	1.3	2 0.0001
13.	0.1326	0.3077	0.1320	-0.0006	1.3	5 —
14.	0.1390	0.3227	0.1385	-0.0005	1.4	5 —
15.	0.1426	0.3312	0.1421	-0.0005	1.4	5 —
16.	0.2000	0.4672	0.2004	+0.0004	2.0	5 0.0002
17.	0.2190	0.5095	0.2186	-0.0004	1.9	10 trace
18.	0.1353	0.3166	0.1358	+0.0005	1.4	10 trace
19.	0.2330	0.5439	0.2334	+0.0004	2.3	10 trace
20.	0.1935	0.4524	0.1941	+0.0006	2.0	10 0.0002
21.	0.1383	0.3213	0.1379	-0.0004	1.4	10 —
22.	0.1509	0.3498	0.1501	-0.0008	1.5	10 —
23.	0.1318	0.3070	0.1317	-0.0001	1.3	10 —
24.	0.2000	0.4649	0.1995	-0.0005	2.0	10 0.0003
25.	0.2000	0.4662	0.2000	—	2.0	10 0.0002

From the above two tables it will be seen that fairly accurate results may be easily obtained, weighing either as zinc ammonium phosphate or as pyrophosphate.

When the proportion of ammonium chloride is high, 15 or 20 grms. for instance, some difficulty will be found in completely washing the precipitate free from chloride, so that, on dissolving the precipitate in nitric acid, a distinct reaction with silver nitrate will be obtained, as was the case in numbers 24 and 25 in Table II. In such cases weighing as pyrophosphate is to be recommended. In all cases where weighing as the double salt is resorted to, it is as well to be assured of the purity of the precipitate by testing as described. As a rule only the very faintest opalescence will be obtained.

The figures given in the seventh column of the two tables represent the total amount of ammonium chloride present after the completion of the reaction,—i. e., the total chlorine calculated as NH_4Cl .

In the extreme right hand column is found the amounts of zinc left in the filtrates. This was detected, and in some cases estimated as follows:—The clear filtrate, after concentration if necessary, was made strongly alkaline with ammonia, then a few drops of ammonium sulphide added, and the whole kept in a warm place for at least twenty-four hours. One-tenth of a m.grm. of zinc gives a perceptible precipitate, in solutions containing ammonium chloride and phosphate, when treated in this way. An attempt to estimate these small amounts was made in the following way:—The liquid was filtered through a Swedish filter-paper, and washed as rapidly as possible, with a little cold distilled water, using the pump to hasten the filtration and so reduce risk of oxidation. The filter-paper with the trace of zinc sulphide on it is then returned to the flask in which the precipitation was effected (taking care to expel by blowing any sulphide vapours from the flask), and an excess of standardised approximately N/100 iodine solution added, together with a drop or two of very dilute hydrochloric acid. After a short time the excess of iodine is titrated back with N/100 sodium thiosulphate. Both volumetric solutions were standardised each time they were used. A similar process, applicable to larger amounts, has quite recently been published by Pouget (*Comptes Rendus*, cxxix., 45—47). No great accuracy is claimed for the figures thus obtained; they are merely intended to approximately indicate the error involved. Where no entry is made as to the amount of zinc in the filtrate, it may be taken as being less than one-tenth of a m.grm., and therefore too small to estimate.

The next point investigated was as to whether the process was equally applicable to sulphates and nitrates.

The following results (Table IV.) clearly indicate that they exert no disturbing influence.

TABLE IV.

No.	Zinc found as taken.	Zinc found as ZnNH_4PO_4 .	Error.	Zinc found as $\text{Zn}_2\text{P}_2\text{O}_7$.	Error.	Ammonium salts.	Zinc in filtrate.
						NH_4NO_3 .	
1.	0.1544	0.1553	+0.0009	0.1553	+0.0009	1	0.0001
2.	0.1109	0.1107	-0.0002	0.1106	-0.0003	2	0.0002
3.	0.1106	0.1100	-0.0006	0.1100	-0.0006	5	—
4.	0.0922	0.0919	-0.0003	0.0919	-0.0003	10	—
						$(\text{NH}_4)_2\text{SO}_4$.	
5.	0.1732	0.1731	-0.0001	0.1729	-0.0003	0.75	0.0002
6.	0.1295	0.1295	—	0.1296	+0.0001	1.5	trace
7.	0.1112	0.1108	-0.0004	0.1108	-0.0004	5	—
8.	0.1358	0.1351	-0.0007	0.1351	-0.0007	10	—

In the above table the results obtained by weighing, both as ZnNH_4PO_4 and as $\text{Zn}_2\text{P}_2\text{O}_7$, are given, and the almost absolute coincidence of the figures obtained by the two methods should be noticed, the extreme difference being two-tenths of a m.grm.

Since, as before stated, it has been considered proved that, under slightly different conditions to those obtaining

in the method described, unless a large amount of ammonium chloride is present in the zinc solution, the precipitate thrown down by an alkaline phosphate does not strictly correspond to the formula ZnNH_4PO_4 , it seemed desirable that other confirmation than test-analyses should be given of the correctness of composition of the precipitate thrown down in the preceding modification. Accordingly the following analyses are given (Table V.) of precipitates obtained from zinc chloride solutions containing varying percentages of ammonium chloride. The precipitates were prepared and treated in exactly the same way as in an actual analysis.

TABLE V.

	ZnNH_4PO_4 from 1 per cent NH_4Cl solution.		ZnNH_4PO_4 from 5 per cent NH_4Cl solution.		ZnNH_4PO_4 from 10 per cent NH_4Cl solution.	
	Theory.	Diff.	Theory.	Diff.	Theory.	Diff.
ZnO ..	45.60	-0.05	45.55	+0.02	45.48	-0.12
NH_3 ..	9.56	+0.01	9.57	+0.02	9.50	-0.06
H_2O ..	5.05	+0.04	5.09	—	5.13	+0.08
P_2O_5 ..	39.79	—	39.75	-0.04	39.89	+0.10
	100.00		100.00		100.00	

The ZnO was determined by observing the weight of precipitate yielded by a known weight of pure zinc oxide. The ammonia was determined by the distillation of about 2 grms. of the precipitate with excess of caustic soda, the evolved NH_3 being absorbed in standard acid, the excess of which was determined by titration in the ordinary way.

The water was determined by subtracting the figure found for the ammonia from the total loss on ignition. The phosphoric acid was taken by difference.

It will be seen that the numbers obtained agree satisfactorily with theory, so that it may be safely assumed that the precipitate is of correct constant composition whether much or little ammonium chloride be present.

It may be of interest to note that an unsuccessful attempt was made to apply Stolba's alkalimetric method for the titration of magnesium ammonium phosphate to the zinc salt. It was also noticed that, if ammonium arsenate be substituted for the phosphate, an uninviting gelatinous precipitate is obtained, quite unlike the double phosphate. The filtrate contained traces of zinc. It was not further investigated.

It is my intention to apply the preceding modification to the estimation of other metals, especially manganese, since Gooch and Austin (*Amer. Journ. Sci.*, 1898, vi., 223) consider that, under ordinary conditions, large amounts of ammonium salts are needed if accurate results are to be obtained.

In conclusion, I wish to express my thanks to Professor Smithells for advice and help in connection with the above paper.—*Zeitschrift f. Anal. Chemie*, xxix., 273—284.

The Yorkshire College, Leeds.

New Method for the Preparation of the Nitromethanes.—V. Auger.—When we react with chloracetate of potassium on nitrite of potassium in concentrated aqueous solution, a lively reaction is produced on heating the mixture, with the disengagement of carbonic acid, and nitromethane distils over:—



Wishing to see if this isolated reaction was susceptible of generalisation, the author first applied it to the bromine derivatives of the acids of the fatty series, and obtained the best results by operating as quickly as possible with the dry materials, and slightly overheating the flask, to prevent the product formed from falling back.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 9.

SYNTHETIC INDIGO.*

By EDWARD S. JOHNSON.

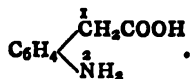
(Continued from p. 90).

I. THE SYNTHESIS OF INDIGO.

THE wonderful investigations of Baeyer and his pupils, which almost exclusively underlie this development, are so remarkable as examples of consistently prosecuted, perspicuous, resourceful, and prolific research, and withal so absolutely essential to a discussion of synthetic indigo, that they will throughout be reviewed somewhat in detail. To this will be added a brief consideration of the chief of more recent technically important syntheses.

1. Baeyer's Syntheses.

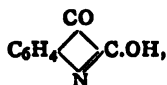
The first of these most interesting formative processes is based upon *o*-amidophenylacetic acid (Baeyer, "Synthese d. Isatine u. d. Indigblaus," *Ber. Deut. Chem. Gesell.*, xi., 1228),—



The anhydride of the acid was shown by Baeyer "Synthese d. Oxindols," *Ber. Deut. Chem. Gesell.*, xi., 584) to be identical with oxindol, one of the compounds of the indigo-group then already known, and in the investigation referred to shown to be—



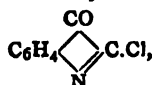
the anhydride of *o*-amidophenylacetic acid. The preparation of isatine,—



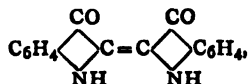
had already been long before realised by the work of Baeyer and Emmerling (*Ber. Deut. Chem. Gesell.*, iii., 514) by the action of phosphorus, phosphorus dichloride, and acetyl chloride upon that substance. The problem therefore consisted in substituting H_2 in the methylene group, CH_2 , of oxindol by oxygen. Great difficulty was experienced in effecting the change, being found impossible by direct oxidation. With the idea of facilitating the desired reaction, chlorine, bromine, &c., were substituted in the methylene group, but all unsuccessfully; by means of nitrous acid, and thus the introduction of the isonitro-group NOH , and reduction, the oxidation was readily and perfectly accomplished:—



The slight yield by the old process for bringing about the last stage of the operation was later (*Ber. Deut. Chem. Gesell.*, xi., 1296; and xii., 456) improved, and the nature of the reaction more fully explained by the use of phosphorus pentachloride. Hereby isatine chloride,—



is formed and by reduction (P , $\text{Zn} + \text{HCl}$, &c. transformed into indigo,—



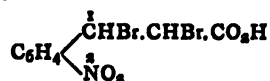
"Thereby for the first time a synthesis of indigo from coal-tar has been effected" (*Ber. Deut. Chem. Gesell.*, xi., 1228). It dates from the year 1878.

Following shortly, in 1880, upon the *o*-amidophenyl-acid synthesis, came the famous investigations starting with *o*-nitrocinnamic acid. They brought to light a series of most peculiar and astonishing substances, the study of which in the main definitely made plain the constitution of indigo.

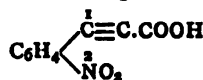
o-Nitrocinnamic acid,—



by addition of bromine is converted into *o*-nitrotribrom-cinnamic acid,—

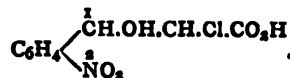


By alkali in the cold, the bromine may be removed in the form of alkali bromide, leaving—

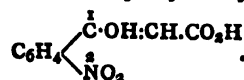


o-nitrophenylpropionic acid. This, with dilute alkali and mild reducing agents, such as milk-sugar, grape-sugar, and xanthates, produces indigo.

With hypochlorites in alkaline solution, *o*-nitrocinnamic acid further yields *o*-nitrophenylchloroacetic acid,—



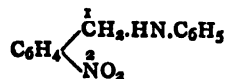
Alcoholic solution of alkali extracts one molecule of hydrochloric acid and *o*-nitrophenylloxacrylic acid,—



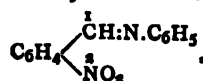
results. Heating at 100° in the presence of a solvent, such as acetic acid or phenol, completes the conversion into indigo. The yield of the latter form of the *o*-nitrocinnamic acid process is slight, while the first mentioned amounts to 70 per cent of the theoretical (Reissert, "Indigo-synthesen," 7).

Baeyer and Drewsen in 1882 published the discovery by them of the formation of indigo from *o*-nitrobenzaldehyde and acetone. This, at that time unaccountable, process was fully investigated by its discoverers. By it indigo is formed when a mixture of the aldehyde and acetone, acetaldehyde, or phenylglyoxalic acid is warmed in the presence of alkali.

The technical starting-point for the preparation of indigo by this method is toluene; this hydrocarbon is nitrated, producing as a main product the *o*-nitro-derivative. This is chlorinated in the methyl-group—a reaction which is only possible to the extent of about 50 per cent without introducing chlorine into the benzene ring also. The separating of the *o*-nitrobenzylchloride formed, from unaffected nitrotoluene, is accomplished by the action of aniline upon the mixture, producing *o*-nitrobenzyl-aniline,—



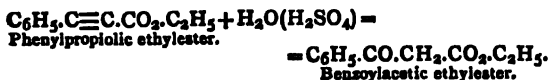
By oxidation, *o*-nitrobenzylideneaniline is formed,—



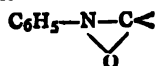
Boiling with hydrochloric acid converts the substance into the aldehyde sought and aniline hydrochloride.

* Proceedings of Engineers' Society of Western Pennsylvania, xvi., No. 3.

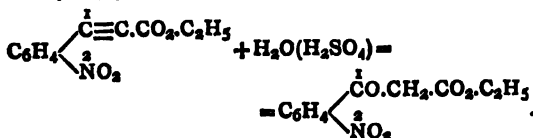
The transformations involved in the conversion of *o*-nitrophenylpropionic acid into indigo are of somewhat complicated nature. Their explanation is based upon certain experimentally derived facts, and assumptions from analogy. Baeyer (*Ber.*, xv., 2705; further, in same connection, compare Reissert, "Indigosynthesen," 19-23, and the literature there referred to) observed that phenylpropionic ethylester by the action of slightly diluted sulphuric acid is converted into benzoylacetic ester,—



It is also a general observation that nitro-groups united to the benzene ring are deprived partially or even wholly, without the use of reducing agents, of oxygen by mobile (easily oxidisable) hydrogen atoms held by carbon attached to the benzene ring in *o*-position to the nitro-group. Moreover, it is known that carbonyl groups (=CO) of aldehyde or ketone nature react with phenylhydroxylamine, cause water to separate, and form compounds which contain the combination of atoms—



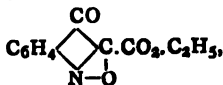
With these facts in mind, analogous transformations may be readily recognised as possible in the case of *o*-nitrophenylpropionic ethylester,—



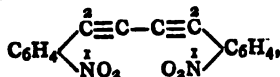
In the *o*-nitrobenzoylacetic ethylester thus formed, intramolecular reduction produces *o*-hydroxylaminobenzoyloxalic ethylester,—



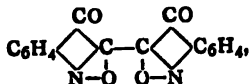
which becomes by anhydriation—



isatogenic ethylester. As to dinitrodiphenyldiacetylene,—

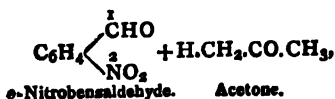


which contains the characteristic group of *o*-nitrophenylpropionic acid twice, it is at once seen how di-isatogen,—

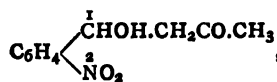


may be derived. By comparison of its formula with that of indigo, it is apparent that the latter is produced by the removal from di-isatogen of two atoms of oxygen and the addition of two atoms of hydrogen; in other words, by reduction. This relationship is in perfect accord with the actual observation.

In the case of the *o*-nitrobenzaldehyde synthesis,—



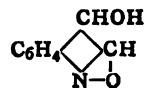
o-nitrophenyllactic ketone,—



is formed as a first phase of the reaction, and is transformed, by internal reduction of the nitro-group, to—



The alkali added to the original mixture separates acetyl (—CO·CH₃) as acetate. This and anhydriation produce—



which may be conceived as changing, by the migration of an hydrogen atom, to—

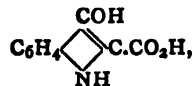


The splitting off of two molecules of water from two molecules of such a combination would result in the formation of indigo.

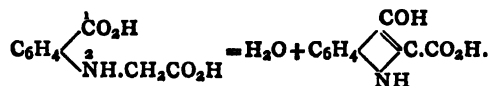
With the last-named synthesis, the following shares the greatest technical importance (Friedlaender, "Fortschritte d. Theerfarbenfabrikation," iv., 1027); it is the—

2. Heumann Synthesis.

In its most recent form, it consists in the preparation of *indoxylic acid*,—



which is readily converted into indigo by alkaline fusion of phenylglycine-*o*-carboxylic acid:—



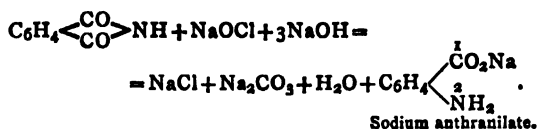
The original raw material for the process is naphthalene, from which, by the action of concentrated sulphuric acid at high temperatures, phthalic anhydride,—



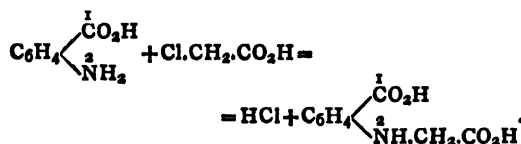
is obtained. From this, phthalimide,—



is prepared by means of ammonia gas. Phthalimide in alkaline solution is converted by sodium hypochlorite into anthranilic acid:—



With monochloroacetic acid, phenylglycine-*o*-carboxylic acid is formed:—



(To be continued).

NOTICES OF BOOKS.

Twenty-fourth Annual Report of Her Majesty's Inspectors of Explosives; being their Report for the Year 1899. London: Darling and Sons. 1900. Pp. 190.

THE department has sustained a loss in the retirement of Colonel Ford, C.B., who succeeded to the post of Chief Inspector on the death of Sir Vivian Majendie. Colonel Ford reached the age limit on the 15th of August, 1899, and was obliged to retire on that date according to the regulations. He is succeeded by Captain J. H. Thomson.

The only modification of the law made during the past year has been an amendment of Order of Secretary of State, No. 3, in regard to the packing of cordite for Government contract.

The growth of trade in explosives during the past year is shown by the great increase in the number of factories, and also by the increased activity in many of the leading factories. The Inspectors, we are glad to see, are able to report that the condition and management of the various factories and magazines visited by them are, in the majority of cases, excellent, and that there has been no falling off in the high standard previously attained, but we may be permitted to ask why the management should not by now be excellent in all cases?

The number of deaths (three) from accidents by fire or explosion in manufacture this year is again below the average for the decade (4.4).

The total number of factories under continuing certificate or license is 151. The total number of factories which have come into existence since the commencement of the Act is 140, but 44 factories have become extinct. The net increase is therefore 96; the net increase for the present year is eight.

The additions to the list of authorised explosives number fifteen, there is one alteration of name, five additions to the authorisable list, and seven alterations in definitions.

The character of the samples taken for examination during the year has been satisfactory. Dr. Dupré's report (Table I.) showing that of 303 samples examined by him only 13 were rejected.

During the year the Inspectors have paid 214 visits to factories, now numbering 151, so that every one has been visited at least once, and nearly half of them a second time. Only one case has occurred during the year in which the Inspector's visit necessitated recourse to judicial proceedings.

The number of accidents reported during the year is 54, causing three deaths and injuring 24 persons. Eight cases of illegal manufacture have come to the Inspector's notice during the year.

There is a considerable falling off in the quantity of nitroglycerin compounds imported during the year, the amount being 798,350 lbs., against 983,000 lbs. in 1898; the amount of non-nitroglycerin explosives imported has also fallen from 177,100 lbs. to 140,882 lbs.

The year has again been one of considerable activity among inventors, and as many as thirteen new explosives have been submitted for examination, or had been left over from the preceding year. The chief point of interest connected with this part of the subject is the fact that for the first time for many years a chlorate mixture has passed successfully through all the tests.

Among the miscellaneous accidents, not immediately connected with blasting, it is significant to notice that the largest number come under the head of "Playing with Detonators," the number having increased from 15 to 22 since the previous year. Of these five occurred to grown up persons, the remainder, however, occurred with detonators which had got into the hands of children. In their last report the Inspectors suggested means by which these accidents could be diminished, and since then rules have been inserted in the Explosives in Coal Mines Order

of July 24th, by which detonators are to be issued only to shot-firers, and to be safely kept by them. These rules only came into force in October last, and it is perhaps too soon to judge of their effect. If, however, the number of such accidents does not decrease during the current year, the Inspectors consider that the alternative suggestion in their last report, viz., that of rendering the detonators less attractive in appearance, should be tried. It is true that these accidents seldom cause loss of life, but it should be remembered that the children who lose fingers or a hand from these explosions are thereby handicapped in the struggle for existence, which in this country is becoming yearly more and more severe.

First Stage Hygiene. By ROBERT A. LYSTER, B.Sc., Lond. London: W. B. Clive, University Tutorial Press. 1900. Pp. 199.

THE increased popularity of the study of hygiene and public health is one of the chief reasons that have led to the publication of this work, and the author believes that it embodies the first attempt that has been made to treat the successive points in logical order. Chapter I. deals with the general build of the body; this is followed by chapters on the blood, air, respiration, ventilation, and foods. In Chapters VI. to X. we have to do with the digestive system, examples of foods, cooking, beverages, &c. There is an interesting chapter, No. XVII., on water supply, in which the general composition and the commoner impurities of water are given, with simple methods for their detection. There is also a useful chapter on accidents and emergencies (No. XV.), describing the best steps to take in cases of cuts, sprains, drowning, suffocation, burns, hysteria, poisoning, &c.

At the ends of those chapters in which the subject matter adapts itself to such treatment, a list of simple experiments, illustrating the work covered by the chapter, has been added. The performance of these experiments will be very useful in helping to fix the facts in the student's memory.

Elementary Practical Chemistry for Schools of Science. By THOMAS CARTWRIGHT, B.A., B.Sc., Lond. London, Edinburgh, and New York: Thomas Nelson and Sons. 1900. Pp. 140.

In this volume the author has attempted to strike the happy mean between the method of discovery and the method of telling, believing that those who would abolish telling and insist on the learner being entirely a discoverer, are as much in the wrong as the teacher who puts his faith in the lecture-room rather than in the laboratory.

The author begins with measuring and weighing, simple blowpipe work, &c., then goes on to experiments with sand and saltpetre, illustrating the operation of separation by means of solution, filtration, and recovery by evaporation. These are followed by experiments with air, oxygen, water, acids, chalk, sulphur, &c., with a few volumetric exercises, and a short chapter on chemical theory.

The book is well arranged and clearly written, and should be found useful by teachers of science for beginners.

Note on Chloronaphthylamine, $C_{10}H_6ClNH_2$ 1.4.—F. Reverdin and P. Crépieux.—On attempting to prepare dichloronaphthylamine by the chlorisation of acetyl- α -naphthylamine, by means of chlorate of soda and hydrochloric acid, the authors obtained a base after saponification, which, when distilled with steam and crystallised several times in ligroin, fused at 98°, while its acetyl derivative had the fusing-point 186.5°. Analysis showed that this base is a monochloronaphthylamine.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 9.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxxiii., No. 9.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 5, July 30, 1900.

Electrolysis of Concentrated Solutions of Hypochlorites.—André Brochet.—The author's experiments show that the electrolysis of a hypochlorite proceeds in the same way as a chloride, and tends towards the same limits.

Gadolinium.—Eug. Demarçay.—(See p. 97).

Diphenylcarbazide as a very Sensitive Reaction for some Metallic Compounds.—P. Cazeneuve.—Diphenylcarbazide, as the author previously showed, is transformed into diphenylcarbazone by losing two atoms of hydrogen under the influence of certain oxides and metallic salts. During this reaction, the diphenylcarbazone formed combined with the metal gives characteristic colours. Salts of copper, mercury, and particularly the per-salts of iron, act in the cold in aqueous solution, forming cuprous, mercurous, and ferrous derivatives of a decidedly intense colour, allowing of the detection of the metal in solutions containing 1/100,000th part of the metallic salt. The diphenylcarbazide, when used as a reagent, should be pure and white; this can be obtained by crystallisation from concentrated acetic acid or pure acetone, and drying the compound at 60°. Copper salts impart a violet colour, mercury a pale blue tint, and iron a peach flower colour, which becomes a dull brown on agitation with ferrocyanide. These colourations are much more sensitive than the ordinary reactions, but they are destroyed by mineral acids and organic acids in excess.

No. 6, August 6, 1900.

Boiling-points of Zinc and Cadmium.—Daniel Berthelot.—The author used specimens of extremely pure zinc, obtained from M. Férent, in which the proportion of impurity was less than one-thousandth part of the whole. Four experiments gave the numbers 924°, 913°, 914°, 922° as the boiling-points of zinc, the mean of these determinations being 920°. A pure specimen of cadmium, also obtained from M. Férent, gave coincident results for three experiments, the boiling-point being 778°.

Atomic Weight of Radio-active Barium.—M^{me}. Curie.—(See p. 98).

Electrolytic Estimation of Cadmium.—Dmitry Balachowsky.—The author obtained, by electrolysis of solutions of bismuth and cadmium, metallic precipitates absolutely free from oxide, very adherent, and easy to wash. The difference in the conditions of precipitation of these two bodies, led the author to devise a means of separation of these two elements which will form the subject of a future paper. He also confirmed the fact that the electrolytic method constitutes an excellent way of obtaining bismuth in a state of absolute purity, as previously shown by A. Classen.

Some New Spectra of Rare Earths.—Eug. Demarçay.—Will be inserted in full.

Blue Oxide of Molybdenum.—Marcel Guichard.—The author succeeds in isolating the blue hydrated oxide of molybdenum in a state of purity. He describes the conditions which are necessary to obtain easily a considerable quantity. His analyses appear to show that it has the formula $\text{MoO}_3 \cdot 4\text{MoO}_3 \cdot 6\text{H}_2\text{O}$. A further investigation of this compound is being made, which will form the subject of a future paper.

A Regulating Furnace for Fixed Temperatures.—A. Gautier.—Already noticed.

Thermic Study of Protocatechic or Dioxibenzoic Acid. Influence of the Phenolic Oxhydriles.—G. Massol.—Commercial protocatechic acid is very impure; the author purified it by fractional crystallisation, transformation into the potash salt, decolouration by bone-black, decomposition of the salt by sulphuric acid, solution of the acid in ether, and fresh crystallisation: it then occurs in beautiful bright yellow, prismatic needles, fusible at 199°. The acidimetric assay made with a normal solution of soda, and without any added indicator, gave a first golden-yellow indication a little before the addition of 1 molecule of alkali (0.88 to 0.90 mol.), then the solution gradually became darker, each drop of alkali produced a rose colouration, and a second wine-red indication was distinctly produced by the second molecule of alkali. On adding phenolphthalein, we first noticed the golden-yellow indication, then the phenolphthalein became red on the addition of 1.5 mols. of the base. With litmus the indication takes place with 2 mols. of alkali. These different reactions show the influence of the phenolic oxhydriles on the general acidity of the molecule; it is also observed that the heats of neutralisation are sensibly lower than those given by the meta- and para-oxybenzoic acids, which contain 1 mol. less of oxhydrile. The author has previously concluded, from his experiments on the oxybenzoic acids, that oxhydrile in the ortho-position (salicylic acid) increases the acidity of benzoic acid, while in the meta- and para-positions it has only a feeble influence (about 0.4 cal.), which should enable us to fix the position of the oxhydrile with regard to the carboxyl. These results are applicable to the dioxibenzoic acids: in protocatechic acid the two oxhydriles are in meta- and para-positions, and their total influence on the carboxyl only increases the heat of formation of the salt by 0.29 cal., while it should have been 2 cal. if one of the phenolic oxhydriles had occupied the ortho-position.

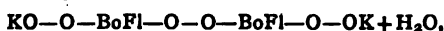
On the Oxide of Diphenylmethanol.—V. Auger.—A controversial paper in which the author concludes that the ether oxide of benzhydrol should keep the formula attributed to it by Linnemann, Zagumeni, Friedel, and Balsohn.

MISCELLANEOUS.

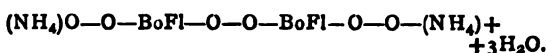
Researches on Graphite.—L. Staudenmaier.—**Graphitic Acid** (from Ceylon graphite).—Crystalline graphitic acid, so-called, is in reality amorphous, and represents a pseudomorphose of crystals of the graphite from which it has been prepared. Chloride of tin transforms it into a brilliant black product, keeping the same crystalline form as the acid, and consequently, of the original graphite. This compound is infusible, only slightly combustible, and resembles graphite entirely. **The Pyro- and Pseudo-graphitic Acids.**—A mixture of 250 c.c. SO_4H_2 and 100 c.c. of water attacks graphitic acid at about 160, giving off CO_2 , and furnishing a brilliant black product, which is pseudomorphous of graphite, very slightly combustible, and easily attacked by oxidising agents; the author calls it pyrographitic acid, although it in no way resembles the product commonly known by this name. It is attacked by nitric acid, giving a series of amorphous red products, and at the same time mellitic acid. Chloric acid attacks it, giving oxidised products, first green, then yellow, resembling graphitic acid, and

its immediate product of combustion, and called by the author pseudo-graphitic acids. These compounds are easily attacked by oxidising agents; they give, with stannous chloride, black products of combustion. The author concludes from his researches that a series of black carbonaceous bodies exists having the appearance of graphite, and a series of green and yellow bodies having the appearance of the graphitic acids. He thinks that graphitic and pseudographitic acids are very complex compounds with a quinonic function, and having carboxylated groups, and that as the complex molecule is attacked the number of these last groups augments at the expense of several quinonic bonds. He is also of the opinion that the study of the graphitic acids can be carried out more easily starting from products with an already simplified molecule similar to that of mellitic acid.—*Berichte*, vol. xxxii., p. 2825.

Fluohyperborates.—P. Mel koff and S. Lordkipanidze.—In a preceding paper the authors have shown that potassic fluoborate, under the influence of peroxide of hydrogen, is converted into a compound corresponding to the empirical formula $\text{Bo}_2\text{K}_4\text{F}_4\text{O}_{11}$. By dissolving this salt in an excess of peroxide of hydrogen, and treating the solution obtained with alcohol, we observe the formation of a viscous precipitate which rapidly changes into a crystalline powder. This latter is soluble in water, and gives off an abundance of oxygen under the action of heat. The new salt, the constitution of which may be expressed by the formula—



ought to be regarded as a fluohyperboric acid, of which the atom of hydrogen has been replaced by the remaining KO. The ammoniacal salt, answering to the formula $(\text{NH}_4)_2\text{Bo}_2\text{F}_2\text{O}_6 + 3\text{H}_2\text{O}$, is obtained by causing equivalent quantities of boric acid and fluoride of ammonium to react in aqueous solution. The solution is treated with an excess of peroxide of hydrogen, and then with ammonia and alcohol. This latter precipitates a fairly stable white crystalline mass, which, in the dry state, gives off a smell of ammonia. Under the influence of heat the aqueous solution of the salt decomposes with the evolution of oxygen and the formation of nitrite of ammonia. From the proportions of boron, ammonia, and active oxygen present in the salt, it is necessary to give it the empirical composition $(\text{NH}_4)_2\text{Bo}_2\text{F}_2\text{O}_6 + 3\text{H}_2\text{O}$ and the extended formula—



As perboric acid has not the property of giving saline compounds with the metallic superoxides, these latter are substituted very easily for the hydrogen atom of fluohyperboric acid.—*Berichte*, vol. xxxii., p. 3510.

A New Method for the Separation of Chlorine and Iodine.—L. Vanino and O. Hauser.—In a previous publication one of the authors has shown that, when we submit halogen derivatives of silver to the influence of alkaline solutions of formic aldehyde, the resistance to reduction increases very rapidly from the chloride to the iodide. On this phenomenon he bases a method for the separation of chlorine from iodine, and he gives the following details of the operations:—The haloid salts are precipitated by means of nitrate of silver, filtered by decantation, and washed with boiling water, taking the necessary precautions to avoid carrying over the least trace of any solid substance. When the washing is complete, the precipitate is treated with 25 c.c. of a solution of 50 grms. of carbonate of potassium in 100 grms. of water and 5 c.c. of 42 per cent formic aldehyde. This is left to stand for some time at the ordinary temperature, and then heated to 30–40° for half-an-hour. As for the particles adhering to the filter, which may have been accidentally carried over during the decantation, they are

also moistened with a few c.c. of the warm mixture of alkaline carbonate and formic aldehyde. Thanks to this treatment, the chloride of silver is converted into metallic silver, while the iodide of silver is not changed. The liquid is again separated by decantation, and the solid portion which remains at the bottom of the beaker is taken up with warm nitric acid. If we then filter again we separate the insoluble iodide of silver from the metallic silver resulting from the reduction of the chloride, and which goes into solution in the dilute nitric acid. There now only remains to weigh the iodide remaining on the filter, and to precipitate the silver contained in the filtrate by means of hydrochloric acid. The author has made a series of estimations, of which the following are two examples:—

		Grm.	Calculated.		Found.
			Grm.	Grm.	
I.	KI	0.2026	AgI ..	0.2865	0.2866
	KCl	0.1074	AgCl ..	0.2064	0.2051
II.	KI	0.1425	AgI ..	0.2015	0.2004
	KCl	0.0988	AgCl ..	0.1899	0.1892

—*Berichte*, vol. xxxii., p. 3615.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

White Rubber.—Can any correspondent oblige by informing me whether there is any preparation of white rubber that will stand heat and boiling, and which can be used in the manufacture of small articles that would have to go through these processes?—**RUBBER.**

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NOTICE IS HEREBY GIVEN that

THE CASTNER-KELLNER ALKALI COMPANY, LIMITED, of 13, Abchurch Lane, in the City of London, have applied for leave to amend the Specification and Drawings of Letters Patent No. 20239 of 1894, granted to Carl Kellner for "Improvements in Electrolytic Apparatus for Decomposing Metallic Salts."

Particulars of the proposed Amendment were set forth in the "Illustrated Official Journal" (Patents) issued on the 22nd of August, 1900.

Any person, or persons, may give notice of opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

(Signed),

C. N. DALTON,
Comptroller-General.

PHILIP M. JUSTICE,
55 & 56, Chancery Lane, London,
Chartered Patent Agent,
For the Applicant.

NOTICE.

The STUDENTS' NUMBER of the **CHEMICAL NEWS** will be published on Friday, September 21st. Gentlemen holding official positions in the Universities, Medical Schools, &c., of the United Kingdom, where Chemistry and Physical Science form a part of the education, who have not yet forwarded the necessary information to our Office for publication in that Number, will confer a favour by sending it with the least possible delay.

Advertisements for this Number should reach the Office not later than Wednesday, 19th September.

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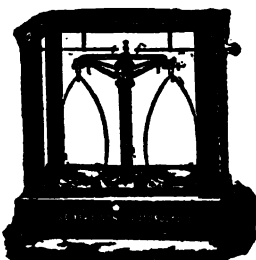
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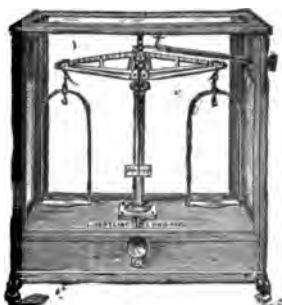
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INAUGURAL ADDRESS OF THE PRESIDENT,
PROF. SIR WILLIAM TURNER, M.B., D.C.L., LL.D., D.Sc.,
F.R.S.

TWENTY-SEVEN years ago the British Association met in Bradford, not at that time raised to the dignity of a City. The meeting was very successful, and was attended by about 2000 persons—a forecast, let us hope, of what we may expect at the present assembly. A distinguished chemist, Professor A. W. Williamson, presided. On this occasion the Association has selected for the presidential chair one whose attention has been given to the study of an important department of biological science. His claim to occupy, however unworthily, the distinguished position in which he has been placed, rests, doubtless, on the fact that, in the midst of the engrossing duties devolving on a teacher in a great University and School of Medicine, he has endeavoured to contribute to the sum of knowledge of the science which he professes. It is a matter of satisfaction to feel that the success of a meeting of this kind does not rest upon the shoulders of the occupant of the presidential chair, but is due to the eminence and active co-operation of the men of science who either preside over or engage in the work of the nine or ten sections into which the Association is divided, and to the energy and ability for organisation displayed by the local Secretaries and Committees. The programme prepared by the general and local officers of the Association shows that no efforts have been spared to provide an ample bill of fare, both in its scientific and social aspects. Members and Associates will, I feel sure, take away from the Bradford Meeting as pleasant memories as did our colleagues of the corresponding Association Française, when, in friendly collaboration at Dover last year, they testified to the common citizenship of the Universal Republic of Science. As befits a leading centre of industry in the great county of York, the applications of science to the industrial arts and to agriculture will form subjects of discussion in the papers to be read at the meeting.

Since the Association was at Dover a year ago, two of its former Presidents have joined the majority. The Duke of Argyll presided at the meeting in Glasgow so far back as 1855. Throughout his long and energetic life he proved himself to be an eloquent and earnest speaker, one who gave to the consideration of public affairs a mind of singular independence, and a thinker and writer in a wide range of human knowledge. Sir J. Wm. Dawson was President at the meeting in Birmingham in 1886. Born in Nova Scotia in 1820, he devoted himself to the study of the Geology of Canada, and became the leading authority on the subject. He took also an active and influential part in promoting the spread of scientific education in the Dominion, and for a number of years he was Principal and Vice-Chancellor of the McGill University, Montreal.

Scientific Method.

Edward Gibbon has told us that diligence and accuracy are the only merits which an historical writer can ascribe

to himself. Without doubt they are fundamental qualities necessary for historical research, but in order to bear fruit they require to be exercised by one whose mental qualities are such as to enable him to analyse the data brought together by his diligence, to discriminate between the false and the true, to possess an insight into the complex motives that determine human action, to be able to recognise those facts and incidents which had exercised either a primary or only a secondary influence on the affairs of nations, or on the thoughts and doings of the person whose character he is depicting.

In scientific research, also, diligence and accuracy are fundamental qualities. By their application new facts are discovered and tabulated, their order of succession is ascertained, and a wider and more intimate knowledge of the processes of nature is acquired. But to decide on their true significance a well-balanced mind and the exercise of prolonged thought and reflection are needed. William Harvey, the father of exact research in physiology, in his memorable work "*De Motu Cordis et Sanguinis*," published more than two centuries ago, tells us of the great and daily diligence which he exercised in the course of his investigations, and the numerous observations and experiments which he collated. At the same time he refers repeatedly to his cogitations and reflections on the meaning of what he had observed, without which the complicated movements of the heart could not have been analysed, their significance determined, and the circulation of the blood in a continuous stream definitely established. Early in the present century, Carl Ernst von Baer, the father of embryological research, showed the importance which he attached to the combination of observation with meditation by placing side by side, on the title-page of his famous treatise "*Ueber Entwicklungsgeschichte der Thiere*" (1828), the words "*Beobachtung und Reflexion*."

Though I have drawn from biological science my illustrations of the need of this combination, it must not be inferred that it applies exclusively to one branch of scientific inquiry; the conjunction influences and determines progress in all the sciences, and when associated with a sufficient touch of imagination, when the power of seeing is conjoined with the faculty of foreseeing, of projecting the mind into the future, we may expect something more than the discovery of isolated facts; their co-ordination and the enunciation of new principles and laws will necessarily follow.

Scientific method consists, therefore, in close observation, frequently repeated so as to eliminate the possibility of erroneous seeing; in experiments checked and controlled in every direction in which fallacies might arise; in continuous reflection on the appearances and phenomena observed, and in logically reasoning out their meaning and the conclusions to be drawn from them. Were the method followed out in its integrity by all who are engaged in scientific investigations, the time and labour expended in correcting errors committed by ourselves or by other observers and experimentalists would be saved, and the volumes devoted annually to scientific literature would be materially diminished in size. Were it applied, as far as the conditions of life admit, to the conduct and management of human affairs, we should not require to be told, when critical periods in our welfare as a nation arise, that we shall muddle through somehow. Recent experience has taught us that wise discretion and careful provision are as necessary in the direction of public affairs as in the pursuit of science, and in both instances, when properly exercised, they enable us to reach with comparative certainty the goal which we strive to attain.

Improvements in Means of Observation.

Whilst certain principles of research are common to all the sciences, each great division requires for its investigation specialised arrangements to insure its progress. Nothing contributes so much to the advancement of

knowledge as improvements in the means of observation, either by the discovery of new adjuncts to research or by a fresh adaptation of old methods. In the industrial arts, the introduction of a new kind of raw material, the recognition that a mixture or blending is often more serviceable than when the substances employed are uncombined, the discovery of new processes of treating the articles used in manufactures, the invention of improved machinery, all lead to the expansion of trade, to the occupation of the people, and to the development of great industrial centres. In science, also, the invention and employment of new and more precise instruments and appliances enable us to appreciate more clearly the significance of facts and phenomena which were previously obscure, and to penetrate more deeply into the mysteries of nature. They mark fresh departures in the history of science, and provide a firm base of support from which a continuous advance may be made and fresh conceptions of nature can be evolved.

It is not my intention, even had I possessed the requisite knowledge, to undertake so arduous a task as to review the progress which has recently been made in the great body of sciences which lie within the domain of the British Association. As my occupation in life has required me to give attention to the science which deals with the structure and organisation of the bodies of man and animals—a science which either includes within its scope or has intimate and widespread relations to comparative anatomy, embryology, morphology, zoology, physiology, and anthropology—I shall limit myself to the attempt to bring before you some of the more important observations and conclusions which have a bearing on the present position of the subject. As this is the closing year of the century it will not, I think, be out of place to refer to the changes which a hundred years have brought about in our fundamental conceptions of the structure of animals. In science, as in business, it is well from time to time to take stock of what we have been doing, so that we may realise where we stand and ascertain the balance to our credit in the scientific ledger.

So far back as the time of the ancient Greeks it was known that the human body and those of the more highly organised animals were not homogeneous, but were built up of parts, the *partes dissimilares* (τὰ ἀνόμοια μέρη) of Aristotle, which differed from each other in form, colour, texture, consistency, and properties. These parts were familiarly known as the bones, muscles, sinews, blood-vessels, glands, brain, nerves, and so on. As the centuries rolled on, and as observers and observations multiplied, a more and more precise knowledge of these parts throughout the Animal Kingdom was obtained, and various attempts were made to classify animals in accordance with their forms and structure. During the concluding years of the last century and the earlier part of the present, the Hunters, William and John, in our country, the Meckels in Germany, Cuvier and St. Hilaire in France, gave an enormous impetus to anatomical studies, and contributed largely to our knowledge of the construction of the bodies of animals. But whilst by these and other observers the most salient, and, if I may use the expression, the grosser characters of animal organisation had been recognised, little was known of the more intimate structure or texture of the parts. So far as could be determined by the unassisted vision, and so much as could be recognised by the use of a simple lens, had indeed been ascertained, and it was known that muscles, nerves, and tendons were composed of threads or fibres, that the blood- and lymph-vessels were tubes, that the parts which we call fasciæ and aponeuroses, were thin membranes, and so on.

Early in the present century Xavier Bichat, one of the most brilliant men of science during the Napoleonic era in France, published his "Anatomie Générale," in which he formulated important general principles. Every animal is an assemblage of different organs, each of which discharges a function, and acting together, each in

its own way, assists in the preservation of the whole. The organs are, as it were, special machines situated in the general building which constitutes the factory or body of the individual. But, further, each organ or special machine is itself formed of tissues which possess different properties. Some, as the blood-vessels, nerves, fibrous tissues, &c., are generally distributed throughout the animal body, whilst others, as bones, muscles, cartilage, &c., are found only in certain definite localities. Whilst Bichat had acquired a definite philosophical conception of the general principles of construction and of the distribution of the tissues, neither he nor his pupil Bérclard was in a position to determine the essential nature of the structural elements. The means and appliances at their disposal and at that of other observers in their generation were not sufficiently potent to complete the analysis.

Attempts were made in the third decennium of this century to improve the methods of examining minute objects by the manufacture of compound lenses, and, by doing away with chromatic and spherical aberration, to obtain, in addition to magnification of the object, a relatively large flat field of vision with clearness and sharpness of definition. When in January, 1830, Joseph Jackson Lister read to the Royal Society his memoir "On some Properties in Achromatic Object-Glasses applicable to the Improvement of Microscopes," he announced the principles on which combinations of lenses could be arranged, which would possess these qualities. By the skill of our opticians, microscopes have now for more than half a century been constructed which, in the hands of competent observers, have influenced and extended biological science with results comparable to those obtained by the astronomer through improvements in the telescope.

In the study of the minute structure of plants and animals the observer has frequently to deal with tissues and organs, most of which possess such softness and delicacy of substance and outline that, even when microscopes of the best construction are employed, the determination of the intimate nature of the tissue, and the precise relation which one element of an organ bears to the other constituent elements, is in many instances a matter of difficulty. Hence additional methods have had to be devised in order to facilitate study and to give precision and accuracy to our observations. It is difficult for one of the younger generation of biologists, with all the appliances of a well-equipped laboratory at his command, with experienced teachers to direct him in his work, and with excellent text-books, in which the modern methods are described, to realise the conditions under which his predecessors worked half a century ago. Laboratories for minute biological research had not been constructed, the practical teaching of histology and embryology had not been organised, experience in methods of work had not accumulated; each man was left to his individual efforts, and had to puzzle his way through the complications of structure to the best of his power. Staining and hardening reagents were unknown. The double-bladed knife invented by Valentin, held in the hand, was the only improvement on the scalpel or razor for cutting thin, more or less translucent slices suitable for microscopic examination; mechanical section-cutters and freezing arrangements had not been devised. The tools at the disposal of the microscopist were little more than knife, forceps, scissors, needles; with acetic acid, glycerine, and Canada balsam as reagents. But in the employment of the newer methods of research care has to be taken, more especially when hardening and staining reagents are used, to discriminate between appearances which are to be interpreted as indicating natural characters, and those which are only artificial productions.

Notwithstanding the difficulties attendant on the study of the more delicate tissues, the compound achromatic microscope provided anatomists with an instrument of great penetrative power. Between the years 1830 and 1850 a number of acute observers applied themselves

with much energy and enthusiasm to the examination of the minute structure of the tissues and organs in plants and animals.

Cell Theory.

It had, indeed, long been recognised that the tissues of plants were to a large extent composed of minute vesicular bodies, technically called cells (Hooke, Malpighi, Grew). In 1831 the discovery was made by the great botanist, Robert Brown, that in many families of plants a circular spot, which he named *areola* or *nucleus*, was present in each cell; and in 1838 M. J. Schleiden published the fact that a similar spot or nucleus was a universal elementary organ in vegetables. In the tissues of animals also structures had begun to be recognised comparable with the cells and nuclei of the vegetable tissues; and in 1839 Theodore Schwann announced the important generalisation that there is one universal principle of development for the elementary part of organisms, however different they may be in appearance, and that this principle is the formation of cells. The enunciation of the fundamental principle that the elementary tissues consisted of cells constituted a step in the progress of biological science, which will for ever stamp the century now drawing to a close with a character and renown equalling those which it has derived from the most brilliant discoveries in the physical sciences. It provided biologists with the visible anatomical units through which the external forces operating on, and the energy generated in, living matter come into play. It dispelled for ever the old mystical idea of the influence exercised by vapours or spirits in living organisms. It supplied the physiologist and pathologist with the specific structures through the agency of which the functions of organisms are discharged in health and disease. It exerted an enormous influence on the progress of practical medicine. A review of the progress of knowledge of the cell may appropriately enter into an address on this occasion.

Structure of Cells.

A cell is a living particle, so minute that it needs a microscope for its examination; it grows in size, maintains itself in a state of activity, responds to the action of stimuli, reproduces its kind, and in the course of time it degenerates and dies.

Let us glance at the structure of a cell to determine its constituent parts and the *role* which each plays in the function to be discharged. The original conception of a cell, based upon the study of the vegetable tissues, was a minute vesicle enclosed by a definite wall, which exercised chemical or metabolic changes on the surrounding material and secreted into the vesicle its characteristic contents. A similar conception was at first also entertained regarding the cells of animal tissues; but as observations multiplied, it was seen that numerous elementary particles, which were obviously in their nature cells, did not possess an enclosing envelope. A wall ceased to have a primary value as a constituent part of a cell, the necessary vesicular character of which therefore could no longer be entertained.

The other constituent parts of a cell are the cell plasma, which forms the body of the cell, and the nucleus embedded in its substance. Notwithstanding the very minute size of the nucleus, which even in the largest cells is not more than 1/500th inch in diameter, and usually is considerably smaller, its almost constant form, its well-defined sharp outline, and its power of resisting the action of strong reagents when applied to the cell, have, from the period of its discovery by Robert Brown, caused histologists to bestow on it much attention. Its structure and chemical composition, its mode of origin, the part which it plays in the formation of new cells, and its function in nutrition and secretion, have been investigated.

When examined under favourable conditions in its passive or resting state, the nucleus is seen to be bounded by a membrane which separates it from the cell-plasma and gives it the characteristic sharp contour. It contains

an apparently structureless nuclear substance, nucleoplasm or enchylema, in which are embedded one or more extremely minute particles called nucleoli, along with a network of exceedingly fine threads or fibres, which in the active living cell play an essential part in the production of new nuclei within the cell. In its chemical composition the nuclear substance consists of albuminous plastin and globulin; and of a special material named nuclein, rich in phosphorus and with an acid reaction. The delicate network within the nucleus consists apparently of the nuclein, a substance which stains with carmine and other dyes, a property which enables the changes, which take place in the network in the production of young cells, to be more readily seen and followed out by the observer.

The mode of origin of the nucleus and the part which it plays in the production of new cells have been the subject of much discussion. Schleiden, whose observations, published in 1838, were made on the cells of plants, believed that within the cell a nucleolus first appeared, and that around it molecules aggregated to form the nucleus. Schwann again, whose observations were mostly made on the cells of animals, considered that an amorphous material existed in organised bodies, which he called *cytoblastema*. It formed the contents of cells, or it might be situated free or external to them. He figuratively compared it to a mother-liquor in which crystals are formed. Either in the *cytoblastema* within the cells or in that situated external to them, the aggregation of molecules around a nucleolus to form a nucleus might occur, and, when once the nucleus had been formed, in its turn it would serve as a centre of aggregation of additional molecules from which a new cell would be produced. He regarded therefore the formation of nuclei and cells as possible in two ways; one within pre-existing cells (*endogenous cell-formation*), the other in a free *blastema* lying external to cells (*free cell-formation*). In animals, he says, the endogenous method is rare, and the customary origin is in an external *blastema*. Both Schleiden and Schwann considered that after the cell was formed the nucleus had no permanent influence on the life of the cell, and usually disappeared.

Under the teaching principally of Henle, the famous Professor of Anatomy in Göttingen, the conception of the free formation of nuclei and cells in a more or less fluid *blastema*, by an aggregation of elementary granules and molecules, obtained so much credence, especially amongst those who were engaged in the study of pathological processes, that the origin of cells within pre-existing cells was to a large extent lost sight of. That a parent cell was requisite for the production of new cells seemed to many investigators to be no longer needed. Without doubt this conception of free cell-formation contributed in no small degree to the belief, entertained by various observers, that the simplest plants and animals might arise, without pre-existing parents, in organic fluids destitute of life, by a process of spontaneous generation—a belief which prevailed in many minds almost to the present day. If, as has been stated, the doctrine of abiogenesis cannot be experimentally refuted, on the other hand it has not been experimentally proved. The burden of proof lies with those who hold the doctrine, and the evidence that we possess is all the other way.

Multiplication of Cells.

Although von Mohl, the botanist, seems to have been the first to recognise (1835) in plants a multiplication of cells by division, it was not until attention was given to the study of the egg in various animals, and to the changes which take place in it, attendant on fertilisation, that in the course of time a much more correct conception of the origin of the nucleus and of the part which it plays in the formation of new cells was obtained. Before Schwann had published his classical memoir in 1839, von Baer and other observers had recognised within the animal ovum the germinal vesicle, which obviously bore to the ovum

the relation of a nucleus to a cell. As the methods of observation improved, it was recognised that, within the developing egg, two vesicles appeared where one only had previously existed, to be followed by four vesicles, then eight, and so on in multiple progression until the ovum contained a multitude of vesicles, each of which possessed a nucleus. The vesicles were obviously cells which had arisen within the original germ-cell or ovum. These changes were systematically described by Martin Barry so long ago as 1839 and 1840 in two memoirs communicated to the Royal Society of London, and the appearance produced, on account of the irregularities of the surface occasioned by the production of new vesicles, was named by him the mulberry-like structure. He further pointed out that the vesicles arranged themselves as a layer within the envelope of the egg or zona pellucida, and that the whole embryo was composed of cells filled with the foundations of other cells. He recognised that the new cells were derived from the germinal vesicle or nucleus of the ovum, the contents of which entered into the formation of the first two cells, each of which had its nucleus, which in its turn resolved itself into other cells, and by a repetition of the process into a greater number. The endogenous origin of new cells within a pre-existing cell, and the process which we now term the segmentation of the yolk, were successfully demonstrated. In a third memoir, published in 1841, Barry definitely stated that young cells originated through division of the nucleus of the parent cell, instead of arising, as a product of crystallisation, in the fluid cytotblastema of the parent cell, or in a blastema situated external to the cell.

In a memoir published in 1842, John Goodsir advocated the view that the nucleus is the reproductive organ of the cell, and that from it, as from a germinal spot, new cells were formed. In a paper, published three years later, on nutritive centres, he described cells, the nuclei of which were the permanent source of successive broods of young cells, which from time to time occupied the cavity of the parent cell. He extended also his observations on the endogenous formation of cells to the cartilage cells in the process of inflammation, and to other tissues undergoing pathological changes. Corroborative observations on endogenous formation were also given by his brother, Harry Goodsir, in 1845. These observations on the part which the nucleus plays by cleavage in the formation of young cells by endogenous development from a parent centre—that an organic continuity existed between a mother cell and its descendants through the nucleus—constituted a great step in advance of the views entertained by Schleiden and Schwann, and showed that Barry and the Goodsirs had a deeper insight into the nature and functions of cells than was possessed by most of their contemporaries, and are of the highest importance when viewed in the light of recent observations.

In 1841 Robert Remak published an account of the presence of two nuclei in the blood corpuscles of the chick and the pig, which he regarded as evidence of the production of new corpuscles by division of the nucleus within a parent cell; but it was not until some years afterwards (1850 to 1855) that he recorded additional observations, and recognised that division of the nucleus was the starting-point for the multiplication of cells in the ovum and in the tissues generally. Remak's view was that the process of cell division began with the cleavage of the nucleolus, followed by that of the nucleus, and that again by cleavage of the body of the cell and of its membrane. Kölliker had previously, in 1843, described the multiplication of nuclei in the ova of parasitic worms, and drew the inference that in the formation of young cells within the egg the nucleus underwent cleavage, and that each of its divisions entered into the formation of a new cell. By these observations, and by others subsequently made, it became obvious that the multiplication of animal cells, either by division of the nucleus within the cell, or by the budding off of a part of the protoplasm of the cell, was to be regarded as a widely spread and probably a

universal process, and that each new cell arose from a parent cell.

Pathological observers were, however, for the most part inclined to consider free cell-formation in a blastema or exudation by an aggregation of molecules, in accordance with the views of Henle, as a common phenomenon. This proposition was attacked with great energy by Virchow in a series of memoirs published in his "Archiv," commencing in Vol. I., 1847, and finally received its death-blow in his published lectures on Cellular Pathology, 1858. He maintained that in pathological structures there was no instance of cell development *de novo*; where a cell existed, there one must have been before. Cell-formation was a continuous development by descent, which he formulated in the expression *omnis cellula e cellula*.

Karyokinesis.

Whilst the descent of cells from pre-existing cells by division of the nucleus during the development of the egg, in the embryos of plants and animals, and in adult vegetable and animal tissues, both in healthy and diseased conditions, had now become generally recognised, the mechanism of the process by which the cleavage of the nucleus took place was for a long time unknown. The discovery had to be deferred until the optician had been able to construct lenses of a higher penetrative power, and the microscopist had learned the use of colouring agents capable of dyeing the finest elements of the tissues. There was reason to believe that in some cases a direct cleavage of the nucleus, to be followed by a corresponding division of the cell into two parts, did occur. In the period between 1870 and 1880 observations were made by Schneider, Strasburger, Bütschli, Fol, van Beneden, and Flemming, which showed that the division of the nucleus and the cell was due to a series of very remarkable changes, now known as indirect nuclear and cell division, or karyokinesis. The changes within the nucleus are of so complex a character that it is impossible to follow them in detail without the use of appropriate illustrations. I shall have to content myself, therefore, with an elementary sketch of the process.

I have previously stated that the nucleus in its passive or resting stage contains a very delicate network of threads or fibres. The first stage in the process of nuclear division consists in the threads arranging themselves in loops, and forming a compact coil within the nucleus. The coil then becomes looser, the loops of threads shorten and thicken, and somewhat later each looped thread splits longitudinally into two portions. As the threads stain when colouring agents are applied to them, they are called chromatin fibres, and the loose coil is the chromosome (Waldeyer).

As the process continues, the investing membrane of the nucleus disappears, and the loops of threads arrange themselves within the nucleus, so that the closed ends of the loops are directed to a common centre, from which the loops radiate outwards, and produce a star-like figure (aster). At the same time clusters of extremely delicate lines appear both in the nucleoplasm and in the body of the cell, named the achromatic figure, which has a spindle-like form with two opposite poles, and stains much more feebly than the chromatic fibres. The loops of the chromatic star then arrange themselves in the equatorial plane of the spindle, and bending round turn their closed ends towards the periphery of the nucleus and the cell.

The next stage marks an important step in the process of division of the nucleus. The two longitudinal portions, into which each looped thread had previously split, now separate from each other, and whilst one part migrates to one pole of the spindle, the other moves to the opposite pole, and the free ends of each loop are directed towards its equator (metakinesis). By this division of the chromatin fibres, and their separation from each other to opposite poles of the spindle, two star-like chromatin figures are produced (dyaster).

Each group of fibres thickens, shortens, becomes surrounded by a membrane, and forms a new or daughter nucleus (dispirem). Two nuclei therefore have arisen within the cell by the division of that which had previously existed, and the expression formulated by Flemming—*omnis nucleus a nucleo*—is justified. Whilst this stage is in course of being completed, the body of the cell becomes constricted in the equatorial plane of the spindle, and, as the constriction deepens, it separates into two parts, each containing a daughter nucleus, so that two nucleated cells have arisen out of a pre-existing cell.

A repetition of the process in each of these cells leads to the formation of other cells, and, although modifications in details are found in different species of plants and animals, the multiplication of cells in the egg and in the tissues generally on similar lines is now a thoroughly established fact in biological science.

In the study of karyokinesis, importance has been attached to the number of chromosomes in the nucleus of the cell. Flemming had seen in the Salamander twenty-four chromosome fibres, which seems to be a constant number in the cells of epithelium and connective tissues. In other cells, again, especially in the ova of certain animals, the number is smaller, and fourteen, twelve, four, and even two only have been described. The theory formulated by Boveri that the number of chromosomes is constant for each species, and that in the karyokinetic figures corresponding numbers are found in homologous cells, seems to be not improbable.

In the preceding description I have incidentally referred to the appearance in the proliferating cell of an achromatic spindle-like figure. Although this was recognised by Fol in 1873, it is only during the last ten or twelve years that attention has been paid to its more minute arrangements and possible signification in cell division.

The pole at each end of the spindle lies in the cell plasma which surrounds the nucleus. In the centre of each pole is a somewhat opaque spot (central body) surrounded by a clear space, which, along with the spot, constitutes the centrosome or the sphere of attraction. From each centrosome extremely delicate lines may be seen to radiate in two directions. One set extends towards the pole at the opposite end of the spindle, and, meeting or coming into close proximity with radiations from it, constitutes the body of the spindle, which, like a perforated mantle, forms an imperfect envelope around the nucleus during the process of division. The other set of radiations is called the polar, and extends in the region of the pole towards the periphery of the cell.

The question has been much discussed whether any constituent part of the achromatic figure, or the entire figure, exists in the cell as a permanent structure in its resting phase; or if it is only present during the process of karyokinesis. During the development of the egg the formation of young cells, by division of the segmentation nucleus, is so rapid and continuous that the achromatic figure, with the centrosome in the pole of the spindle, is a readily recognisable object in each cell. The polar and spindle-like radiations are in evidence during karyokinesis, and have apparently a temporary endurance and function. On the other hand, van Beneden and Boveri were of opinion that the central body of the centrosome did not disappear when the division of the nucleus came to an end, but that it remained as a constituent part of a cell lying in the cell plasma near to the nucleus. Flemming has seen the central body with its sphere in leucocytes, as well as in epithelial cells and those of other tissues. Subsequently Heidenhain and other histologists have recorded similar observations. It would seem, therefore, as if there were reason to regard the centrosome, like the nucleus, as a permanent constituent of a cell. This view, however, is not universally entertained. If not always capable of demonstration in the resting stage of a cell, it is doubtless to be regarded as potentially present, and ready to assume, along with the radiations, a characteristic

appearance when the process of nuclear division is about to begin.

One can scarcely regard the presence of so remarkable an appearance as the achromatic figure without associating with it an important function in the economy of the cell. As from the centrosome at the pole of the spindle both sets of radiations diverge, it is not unlikely that it acts as a centre or sphere of energy and attraction. By some observers the radiations are regarded as substantive fibrillar structures, elastic or even contractile in their properties. Others, again, look upon them as morphological expressions of chemical and dynamical energy in the protoplasm of the cell body. On either theory we may assume that they indicate an influence, emanating, it may be, from the centrosome, and capable of being exercised both on the cell plasma and on the nucleus contained in it. On the contractile theory, the radiations which form the body of the spindle either by actual traction of the supposed fibrillæ or by their pressure on the nucleus which they surround, might impel during karyokinesis the dividing chromosome elements towards the poles of the spindle, to form there the daughter nuclei. On the dynamical theory, the chemical and physical energy in the centrosome might influence the cell plasma and the nucleus, and attract the chromosome elements of the nucleus to the poles of the spindle. The radiated appearance would therefore be consequent and attendant on the physico-chemical activity of the centrosome. One or other of these theories may also be applied to the interpretation of the significance of the polar radiations.

Cell Plasma.

In the cells of plants, in addition to the cell wall, the cell body and the cell juice require to be examined. The material of the cell body, or the cell contents, was named by von Mohl (1846) protoplasm, and consisted of a colourless tenacious substance, which partly lined the cell wall (primordial utricle), and partly traversed the interior of the cell as delicate threads enclosing spaces (vacuoles) in which the cell juice was contained. In the protoplasm the nucleus was embedded. Nägeli, about the same time, had also recognised the difference between the protoplasm and the other contents of vegetable cells, and had noticed its nitrogenous composition.

Though the analogy with a closed bladder or vesicle could no longer be sustained in the animal tissues, the name "cell" continued to be retained for descriptive purposes, and the body of the cell was spoken of as a more or less soft substance enclosing a nucleus (Leydig). In 1861 Max Schultze adopted for the substance forming the body of the animal cell the term "protoplasm." He defined a cell to be a particle of protoplasm in the substance of which a nucleus was situated. He regarded the protoplasm, as indeed had previously been pointed out by the botanist, Unger, as essentially the same as the contractile sarcode which constitutes the body and pseudopodia of the Amoeba and other Rhizopoda. As the term "protoplasm," as well as that of "bioplasm" employed by Lionel Beale in a somewhat similar though not precisely identical sense, involves certain theoretical views of the origin and function of the body of the cell, it would be better to apply to it the more purely descriptive term, "cytoplasm" or "cell plasma."

Schultze defined protoplasm as a homogeneous, glassy, tenacious material, of a jelly-like or somewhat firmer consistency, in which numerous minute granules were embedded. He regarded it as the part of the cell especially endowed with vital energy, whilst the exact function of the nucleus could not be defined. Based upon this conception of the jelly-like character of protoplasm, the idea for a time prevailed that a structureless, dimly granular, jelly or slime destitute of organisation, possessed great physiological activity, and was the medium through which the phenomena of life were displayed.

More accurate conceptions of the nature of the cell plasma soon began to be entertained. Brücke recognised

that the body of the cell was not simple, but had a complex organisation. Flemming observed that the cell plasma contained extremely delicate threads, which frequently formed a network, the interspaces of which were occupied by a more homogeneous substance. Where the threads crossed each other, granular particles (*mikrosomen*) were situated. Bütschli considered that he could recognise in the cell plasma a honeycomb-like appearance, as if it consisted of excessively minute chambers in which a homogeneous more or less fluid material was contained. The polar and spindle-like radiations visible during the process of karyokinesis, which have already been referred to, and the presence of the centrosome, possibly even during the resting stage of the cell, furnished additional illustrations of differentiation within the cell plasma. In many cells there appears also to be a difference in the character of the cell plasma which immediately surrounds the nucleus, and that which lies at and near the periphery of the cell. The peripheral part (*ektoplasma*) is more compact, and gives a definite outline to the cell, although not necessarily differentiating into a cell membrane. The inner part (*endoplasma*) is softer, and is distinguished by a more distinct granular appearance, and by containing the products specially formed in each particular kind of cell during the nutritive process.

By the researches of numerous investigators on the internal organisation of cells in plants and animals, a large body of evidence has now been accumulated, which shows that both the nucleus and the cell plasma consist of something more than a homogeneous, more or less viscid, slimy material. Recognisable objects in the form of granules, threads, or fibres can be distinguished in each. The cell plasma and the nucleus respectively are therefore not of the same constitution throughout, but possess polymorphic characters, the study of which in health and the changes produced by disease will for many years to come form important matters for investigation.

Function of Cells.

It has already been stated that, when new cells arise within pre-existing cells, division of the nucleus is associated with cleavage of the cell plasma, so that it participates in the process of new cell-formation. Undoubtedly, however, its rôle is not limited to this function. It also plays an important part in secretion, nutrition, and the special functions discharged by the cells in the tissues and organs of which they form morphological elements.

Between 1838 and 1842 observations were made which showed that cells were constituent parts of secreting glands and mucous membranes (Schwann, Henle). In 1842 John Goodsir communicated to the Royal Society of Edinburgh a memoir on secreting structures, in which he established the principle that cells are the ultimate secreting agents; he recognised in the cells of the liver, kidney, and other organs the characteristic secretion of each gland. The secretion was, he said, situated between the nucleus and the cell wall. At first he thought that, as the nucleus was the reproductive organ of the cell, the secretion was formed in the interior of the cell by the agency of the cell wall; but three years later he regarded it as a product of the nucleus. The study of the process of spermatogenesis by his brother, Harry Goodsir, in which the head of the spermatozoon was found to correspond with the nucleus of the cell in which the spermatozoon arose, gave support to the view that the nucleus played an important part in the genesis of the characteristic product of the gland cell.

The physiological activity of the cell plasma and its complex chemical constitution soon after began to be recognised. Some years before Max Schultz had published his memoirs on the characters of protoplasm, Brücke had shown that the well known changes in tint in the skin of the *Chamæleon* were due to pigment granules situated in cells in the skin, which were sometimes diffused throughout the cells, at others concentrated in the centre. Similar observations on the skin of the frog

were made in 1854 by von Wittich and Harless. The movements were regarded as due to contraction of the cell wall on its contents. In a most interesting paper on the pigmentary system in the frog, published in 1858, Lord Lister demonstrated that the pigment granules moved in the cell plasma, by forces resident within the cell itself, acting under the influence of an external stimulant, and not by a contractility of the wall. Under some conditions the pigment was attracted to the centre of the cell, when the skin became pale; under other conditions the pigment was diffused throughout the body and the branches of the cell, and gave to the skin a dark colour. It was also experimentally shown that a potent influence over these movements was exercised by the nervous system.

The study of the cells of glands engaged in secretion, even when the secretion is colourless, and the comparison of their appearance when secretion is going on with that seen when the cells are at rest, have shown that the cell plasma is much more granular and opaque, and contains larger particles during activity than when the cell is passive; the body of the cell swells out from an increase in the contents of its plasma, and chemical changes accompany the act of secretion. Ample evidence, therefore, is at hand to support the position taken by John Goodsir, nearly sixty years ago, that secretions are formed within cells, and lie in that part of the cell which we now say consists of the cell plasma; that each secreting cell is endowed with its own peculiar property, according to the organ in which it is situated, so that bile is formed by the cells in the liver, milk by those in the mamma, and so on.

Intimately associated with the process of secretion is that of nutrition. As the cell plasma lies at the periphery of a cell, and as it is, alike both in secretion and nutrition, brought into closest relation with the surrounding medium, from which the pabulum is derived, it is necessarily associated with nutritive activity. Its position enables it to absorb nutritive material directly from without, and in the process of growth it increases in amount by interstitial changes and additions throughout its substance, and not by mere accretions on its surface.

Hitherto I have spoken of a cell as a unit, independent of its neighbours as regards its nutrition, and the other functions which it has to discharge. The question has, however, been discussed, whether in a tissue composed of cells closely packed together, cell plasma may not give origin to processes or threads which are in contact or continuous with corresponding processes of adjoining cells, and that cells may therefore, to some extent, lose their individuality in the colony of which they are members. Appearances were recognised between 1863 and 1870 by Schrön and others in the deeper cells of the epidermis, and of some mucous membranes which gave sanction to this view, and it seems possible, through contact or continuity of threads connecting a cell with its neighbours, that cells may exercise a direct influence on each other.

Nägeli, the botanist, as the foundation of a mechanico-physiological theory of descent, considered that in plants a network of cell plasma, named by him *idio-plasm*, extended throughout the whole of the plant, forming its specific molecular constitution, and that growth and activity were regulated by its conditions of tension and movements (1884).

The study of the structure of plants, with special reference to the presence of an intercellular network, has for some years been pursued by Walter Gardiner (1882-97), who has demonstrated threads of cell plasma protruding through the walls of vegetable cells, and continuous with similar threads from adjoining cells. Structurally, therefore, a plant may be conceived to be built up of a nucleated cytoplasmic network, each nucleus with the branching cell plasma surrounding it being a centre of activity. On this view a cell would retain, to some extent, its individuality, though, as Gardiner contends, the connecting

threads would be the medium for the conduction of impulses and of food from a cell to those which lie around it. For the plant cell, therefore, as has long been accepted in the animal cell, the wall is reduced to a secondary position, and the active constituent is the nucleated cell plasm. It is not unlikely that the absence of a controlling nervous system in plants requires the plasm of adjoining cells to be brought into more immediate contact and continuity than is the case with the generality of animal cells, so as to provide a mechanism for harmonising the nutritive and other functional processes in the different areas in the body of the plant. In this particular it is of interest to note that the epithelial tissues in animals, where somewhat similar connecting arrangements occur, are only indirectly associated with the nervous and vascular systems, so that, as in plants, the cells may require, for nutritive and other purposes, to act and re-act directly on each other.

Nerve Cells.

Of recent years great attention has been paid to the intimate structure of nerve cells, and to the appearance which they present when in the exercise of their functional activity. A nerve cell is not a secreting cell; that is, it does not derive from the blood or surrounding fluid a pabulum, which it elaborates into a visible, palpable secretion, characteristic of the organ of which the cell is a constituent element, to be in due course discharged into a duct which conveys the secretion out of the gland. Nerve cells, through the metabolic changes which take place in them in connection with their nutrition, are associated with the production of the form of energy specially exhibited by animals which possess a nervous system, termed nerve energy. It has long been known that every nerve cell has a body in which a relatively large nucleus is situated. A most important discovery was the recognition that the body of every nerve cell had one or more processes growing out from it. More recently it has been proved, chiefly through the researches of Schultze, His, Golgi, and Ramon y Cajal, that at least one of the processes, the axon of the nerve cell, is continued into the axial cylinder of a nerve fibre, and that in the multipolar nerve cell the other processes, or dendrites, branch and ramify for some distance away from the body. A nerve fibre is therefore an essential part of the cell with which it is continuous, and the cell, its processes, the nerve fibre, and the collaterals which arise from the nerve fibre collectively form a neuron or structural nerve unit (Waldeyer). The nucleated body of the nerve cell is the physiological centre of the unit.

The cell plasm occupies both the body of the nerve cell and its processes. The intimate structure of the plasm has, by improved methods of observation introduced during the last eight years by Nissl, and conducted on similar lines by other investigators, become more definitely understood. It has been ascertained that it possesses two distinct characters which imply different structures. One of these stains deeply on the addition of certain dyes, and is named chromophile or chromatic substance; the other, which does not possess a similar property, is the achromatic network. The chromophile is found in the cell body and the dendritic processes, but not in the axon. It occurs in the form of granular particles, which may be scattered throughout the plasm, or aggregated into little heaps which are elongated or fusiform in shape, and appear as distinct coloured particles or masses. The achromatic network is found in the cell body and the dendrites, and is continued also into the axon, where it forms the axial cylinder of the nerve fibre. It consists apparently of delicate threads or fibrillæ, in the meshes of which a homogeneous material, such as is found in cell plasm generally, is contained. In the nerve cells, as in other cells, the plasm is, without doubt, concerned in the process of cell nutrition. The achromatic fibrillæ exercise an important influence on the axon or nerve fibre with which they are continuous, and probably they conduct the nerve impulses which manifest themselves in the form of

nerve energy. The dendritic processes of a multipolar nerve cell ramify in close relation with similar processes branching from other cells in the same group. The collaterals and the free end of the axon fibre process branch and ramify in association with the body of a nerve cell or of its dendrites. We cannot say that these parts are directly continuous with each other to form an intercellular network, but they are apparently in apposition, and through contact exercise influence one on the other in the transmission of nerve impulses.

There is evidence to show that in the nerve cell the nucleus, as well as the cell plasm, is an effective agent in nutrition. When the cell is functionally active, both the cell body and the nucleus increase in size (Vas, G. Mann, Lugaro); on the other hand, when nerve cells are fatigued through excessive use, the nucleus decreases in size and shrivels; the cell plasm also shrinks, and its coloured or chromophile constituent becomes diminished in quantity, as if it had been consumed during the prolonged use of the cell (Hodge, Mann, Lugaro). It is interesting also to note that in hibernating animals in the winter season, when their functional activity is reduced to a minimum, the chromophile in the plasm of the nerve cells is much smaller in amount than when the animal is leading an active life in the spring and summer (G. Levi).

When a nerve cell has attained its normal size, it does not seem to be capable of reproducing new cells in its substance by a process of karyokinesis, such as takes place when young cells arise in the egg and in the tissues generally. It would appear that nerve cells are so highly specialised in their association with the evolution of nerve energy, that they have ceased to have the power of reproducing their kind, and the metabolic changes, both in cell plasm and nucleus, are needed to enable them to discharge their very peculiar function. Hence it follows that when a portion of the brain or other nerve-centre is destroyed, the injury is not repaired by the production of fresh specimens of their characteristic cells, as would be the case in injuries to bones and tendons.

In our endeavours to differentiate the function of the nucleus from that of the cell plasm, we should not regard the former as concerned only in the production of young cells, and the latter as the exclusive agent in growth, nutrition, and, where gland cells are concerned, in the formation of their characteristic products. As regards cell reproduction also, though the process of division begins in the nucleus in its chromosome constituents, the achromatic figure in the cell plasm undoubtedly plays a part, and the cell plasm itself ultimately undergoes cleavage.

A few years ago the tendency amongst biologists was to ignore or attach but little importance to the physiological use of the nucleus in the nucleated cell, and to regard the protoplasm as the essential and active constituent of living matter; so much so, indeed, was this the case that independent organisms regarded as distinct species were described as consisting of protoplasm destitute of a nucleus; also that scraps of protoplasm separated from larger nucleated masses could, when isolated, exhibit vital phenomena. There is reason to believe that a fragment of protoplasm, when isolated from the nucleus of a cell, though retaining its contractility and capable of nourishing itself for a short time, cannot increase in amount, act as a secreting structure, or reproduce its kind; it soon loses its activity, withers, and dies. In order that these qualities of living matter should be retained, a nucleus is, by most observers, regarded as necessary (Nussbaum, Gruber, Haberlandt, Korschelt), and for the complete manifestation of vital activity both nucleus and cell plasm are required.

Bacteria.

The observations of Cohn, made about thirty years ago, and those of De Bary shortly afterwards, brought into notice a group of organisms, to which the name "bacterium" or "microbe" is given. They were seen to vary in shape; some were rounded specks called cocci, others were straight rods called bacilli, others were curved

or spiral rods, vibrios or spirillæ. All were characterised by their extreme minuteness, and required for their examination the highest powers of the best microscopes. Many bacteria measure in their least diameter not more than $1/25,000$ th of an inch, one-tenth the diameter of a human white blood corpuscle. Through the researches of Pasteur, Lord Lister, Koch, and other observers, bacteria have been shown to play an important part in nature. They exercise a very remarkable power over organic substances, especially those which are complex in chemical constitution, and can resolve them into simpler combinations. Owing to this property, some bacteria are of great economic value, and without their agency many of our industries could not be pursued; others, again, and these are the most talked of, exercise a malignant influence in the production of the most deadly diseases which afflict man and the domestic animals.

Great attention has been given to the structure of bacteria and to their mode of propagation. When examined in the living state and magnified about 2000 times, a bacterium appears as a homogeneous particle, with a sharp definite outline, though a membranous envelope or wall, distinct from the body of the bacterium, cannot at first be recognised; but when treated with reagents a membranous envelope appears, the presence of which, without doubt, gives precision of form to the bacterium. The substance within the membrane contains granules which can be dyed with colouring agents. Owing to their extreme minuteness, it is difficult to pronounce an opinion on the nature of the chromatin granules and the substance in which they lie. Some observers regard them as nuclear material, invested by only a thin layer of protoplasm, on which view a bacterium would be a nucleated cell. Others consider the bacterium as formed of protoplasm containing granules capable of being coloured, which are a part of the protoplasm itself, and not a nuclear substance. On the latter view, bacteria would consist of cell plasma enclosed in a membrane and destitute of a nucleus. Whatever be the nature of the granule-containing material, each bacterium is regarded as a cell, the minutest and simplest living particle capable of an independent existence that has yet been discovered.

Bacteria cells, like cells generally, can reproduce their kind. They multiply by simple fission, probably with an ingrowth of the cell wall, but without the karyokinetic phenomena observed in nucleated cells. Each cell gives rise to two daughter cells, which may for a time remain attached to each other and form a cluster or a chain, or they may separate and become independent isolated cells. The multiplication, under favourable conditions of light, air, temperature, moisture and food, goes on with extraordinary rapidity, so that in a few hours many thousands new individuals may arise from a parent bacterium.

Connected with the life-history of a bacterium cell is the formation in its substance, in many species and under certain conditions, of a highly refractile shiny particle called a spore. At first sight a spore seems as if it were the nucleus of the bacterium cell, but it is not always present when multiplication by cleavage is taking place, and when present it does not appear to take part in the fission. On the other hand, a spore, from the character of its envelope, possesses great power of resistance, so that dried bacteria, when placed in conditions favourable to germination, can through their spores germinate and resume an active existence. Spore formation seems, therefore, to be a provision for continuing the life of the bacterium under conditions which, if spores had not formed, would have been the cause of its death.

The time has gone by to search for the origin of living organisms by a spontaneous aggregation of molecules in vegetable or other infusions, or from a layer of formless primordial slime diffused over the bed of the ocean. Living matter during our epoch has been, and continues to be, derived from pre-existing living matter, even when it possesses the simplicity of structure of a bacterium, and the morphological unit is the cell.

Development of the Egg.

As the future of the entire organism lies in the fertilised egg cell, we may now briefly review the arrangements, consequent on the process of segmentation, which lead to the formation let us say in the egg of a bird, of the embryo or young chick.

In the latter part of the last century, C. F. Wolff observed that the beginning of the embryo was associated with the formation of layers, and in 1817 Pander demonstrated that in the hen's egg at first one layer, called mucous, appeared, then a second or serous layer, to be followed by a third, intermediate or vascular layer. In 1828 von Baer amplified our knowledge in his famous treatise, which from its grasp of the subject created a new epoch in the science of embryology. It was not, however, until the discovery by Schwann of cells as constant factors in the structure of animals and in their relation to development that the true nature of these layers was determined. We now know that each layer consists of cells, and that all the tissues and organs of the body are derived from them. Numerous observers have devoted themselves for many years to the study of each layer, with the view of determining the part which it takes in the formation of the constituent parts of the body, more especially in the higher animals, and the important conclusion has been arrived at that each kind of tissue invariably arises from one of these layers and from no other.

The layer of cells which contributes, both as regards the number and variety of the tissues derived from it, most largely to the formation of the body is the middle layer or mesoblast. From it the skeleton, the muscles, and other locomotor organs, the true skin, the vascular system, including the blood, and other structures which I need not detail, take their rise. From the inner layer of cells or hypoblast, the principal derivatives are the epithelial lining of the alimentary canal, and of the glands which open into it, and the epithelial lining of the air passages. The outer or epiblast layer of cells gives origin to the epidermis or scarf skin and to the nervous system. It is interesting to note that from the same layer of the embryo arise parts so different in importance as the cuticle—a mere protecting structure, which is constantly being shed when the skin is subjected to the friction of a towel or the clothes—and the nervous system, including the brain, the most highly differentiated system in the animal body. How completely the cells from which they are derived had diverged from each other in the course of their differentiation in structure and properties is shown by the fact that the cells of the epidermis are continually engaged in reproducing new cells to replace those which are shed, whilst the cells of the nervous system have apparently lost the power of reproducing their kind.

In the early stage of the development of the egg, the cells in a given layer resemble each other in form, and, as far as can be judged from their appearance, are alike in structure and properties. As the development proceeds, the cells begin to show differences in character, and in the course of time the tissues which arise in each layer differentiate from each other and can be readily recognised by the observer. To use the language of von Baer, a generalised structure has become specialised, and each of the special tissues produced exhibits its own structure and properties. These changes are coincident with a rapid multiplication of the cells by cleavage, and thus increase in size of the embryo accompanies specialisation of structure. As the process continues, the embryo gradually assumes the shape characteristic of the species to which its parents belonged, until at length it is fit to be born and to assume a separate existence.

The conversion of cells, at first uniform in character, into tissues of a diverse kind is due to forces inherent in the cells in each layer. The cell plasma plays an active though not an exclusive part in the specialisation; for as the nucleus influences nutrition and secretion, it acts as a factor in the differentiation of the tissues. When tissues so diverse in character as muscular fibre, cartilage, fibrous

tissues, and bone arise from the cells of the middle or mesoblast layer, it is obvious that, in addition to the morphological differentiation affecting form and structure, a chemical differentiation affecting composition also occurs, as the result of which a physiological differentiation takes place. The tissues and organs become fitted to transform the energy derived from the food into muscular energy, nerve energy, and other forms of vital activity. Corresponding differentiations also modify the cells of the outer and inner layers. Hence the study of the development of the generalised cell layers in the young embryo enables us to realise how all the complex constituent parts of the body in the higher animals and in man are evolved by the process of differentiation from a simple nucleated cell—the fertilised ovum. A knowledge of the cell and of its life-history is therefore the foundation-stone on which biological science in all its departments is based.

If we are to understand by an organ in the biological sense a complex body capable of carrying on a natural process, a nucleated cell is an organ in its simplest form. In a unicellular animal or plant such an organ exists in its most primitive stage. The higher plants and animals again are built up of multitudes of these organs, each of which, whilst having its independent life, is associated with the others, so that the whole may act in unison for a common purpose. As in one of your great factories each spindle is engaged in twisting and winding its own thread, it is at the same time intimately associated with the hundreds of other spindles in its immediate proximity, in the manufacture of the yarn from which the web of cloth is ultimately to be woven.

It has taken more than fifty years of hard and continuous work to bring our knowledge of the structure and development of the tissues and organs of plants and animals up to the level of the present day. Amidst the host of names of investigators, both at home and abroad, who have contributed to its progress, it may seem invidious to particularise individuals. There are, however, a few that I cannot forbear to mention, whose claim to be named on such an occasion as this will be generally conceded.

Botanists will, I think, acknowledge Wilhelm Hofmeister as a master in morphology and embryology, Julius von Sachs as the most important investigator in vegetable physiology during the last quarter of a century, and Strasburger as a leader in the study of the phenomena of nuclear division.

The researches of the veteran Professor of Anatomy in Würzburg, Albert von Kölliker, have covered the entire field of animal histology. His first paper, published fifty-nine years ago, was followed by a succession of memoirs and books on human and comparative histology and embryology, and culminated in his great treatise on the structure of the brain, published in 1896. Notwithstanding the weight of more than eighty years, he continues to prosecute histological research, and has published the results of his latest, though let us hope not his last, work during the present year.

Amongst our own countrymen, and belonging to the generation which has almost passed away, was William Bowman. His investigations between 1840 and 1850 on the mucous membranes, muscular fibre, and the structure of the kidney, together with his researches on the organs of sense, were characterised by a power of observation and of interpreting difficult and complicated appearances which has made his memoirs on these subjects landmarks in the history of histological inquiry.

Of the younger generation of biologists Francis Maitland Balfour, whose early death is deeply deplored as a loss to British Science, was one of the most distinguished. His powers of observation and philosophic perception gave him a high place as an original enquirer, and the charm of his personality—for charm is not the exclusive possession of the fairer sex—endeared him to his friends.

General Morphology.

Along with the study of the origin and structure of the tissues of organised bodies, much attention has been given during the century to the parts or organs in plants and animals, with the view of determining where and how they take their rise, the order of their formation, the changes which they pass through in the early stages of development, and their relative positions in the organism to which they belong. Investigations on these lines are spoken of as morphological, and are to be distinguished from the study of their physiological or functional relations, though both are necessary for the full comprehension of the living organism.

The first to recognise that morphological relations might exist between the organs of a plant, dissimilar as regards their function, was the poet Goethe, whose observations, guided by his imaginative faculty, led him to declare that the calyx, corolla, and other parts of a flower, the scales of a bulb, &c., were metamorphosed leaves, a principle generally accepted by botanists, and indeed extended to other parts of a plant, which are referred to certain common morphological forms although they exercise different functions. Goethe also applied the same principle in the study of the skeletons of vertebrate animals, and he formed the opinion that the spinal column and the skull were essentially alike in construction, and consisted of vertebrae, an idea which was also independently conceived and advocated by Oken.

The anatomist who in our country most strenuously applied himself to the morphological study of the skeleton was Richard Owen, whose knowledge of animal structure, based upon his own dissections, was unrivalled in range and variety. He elaborated the conception of an ideal, archetype vertebrate form, which had no existence in nature, and to which, subject to modifications in various directions, he considered all vertebrate skeletons might be referred. Owen's observations were conducted to a large extent on the skeletons of adult animals, of the knowledge of which he was a master. As in the course of development modifications in shape and in the relative position of parts not unfrequently occur, and their original character and place of origin become obscured, it is difficult, from the study only of adults, to arrive at a correct interpretation of their morphological significance. When the changes which take place in the skull during its development, as worked out by Reichert and Rathke, became known and their value had become appreciated, many of the conclusions arrived at by Owen were challenged and ceased to be accepted. It is, however, due to that eminent anatomist to state, from my personal knowledge of the condition of anatomical science in this country fifty years ago, that an enormous impulse was given to the study of comparative morphology by his writings, and by the criticisms to which they were subjected.

There can be no doubt that generalised arrangements do exist in the early embryo which, up to a certain stage, are common to animals that in their adult condition present diverse characters and out of which the forms special to different groups are evolved. As an illustration of this principle, I may refer to the stages of development of the great arteries in the bodies of vertebrate animals. Originally, as the observations of Rathke have taught us, the main arteries are represented by pairs of symmetrically arranged vascular arches, some of which enlarge and constitute the permanent arteries in the adult, whilst others disappear. The increase in size of some of these arches, and the atrophy of others, are so constant for different groups that they constitute anatomical features as distinctive as the modifications in the skeleton itself. Thus in mammals the fourth vascular arch on the left side persists, and forms the arch of the aorta; in birds the corresponding part of the aorta is an enlargement of the fourth right arch, and in reptiles both arches persist to form the great artery. That this original symmetry exists also in man we know from the fact that now and

again his body, instead of corresponding with the mammalian type, has an aortic arch like that which is natural to the bird, and in rarer cases even to the reptile. A type form common to the vertebrata does therefore in such cases exist, capable of evolution in more than one direction.

The reputation of Thomas Henry Huxley as a philosophic comparative anatomist rests largely on his early perception of, and insistence on, the necessity of testing morphological conclusions by a reference to the development of parts and organs, and by applying this principle in his own investigations. The principle is now so generally accepted by both botanists and anatomists that morphological definitions are regarded as depending essentially on the successive phases of the development of the parts under consideration.

The morphological characters exhibited by a plant or animal tend to be hereditarily transmitted from parents to offspring, and the species is perpetuated. In each species the evolution of an individual, through the developmental changes in the egg, follows the same lines in all the individuals of the same species, which possess therefore in common the features called specific characters. The transmission of these characters is due, according to the theory of Weismann, to certain properties possessed by the chromosome constituents of the segmentation nucleus in the fertilised ovum, named by him the germ-plasm, which is continued from one generation to another, and impresses its specific character on the egg and on the plant or animal developed from it.

As has already been stated, the special tissues which build up the bodies of the more complex organisms are evolved out of cells which are at first simple in form and appearance. During the evolution of the individual, cells become modified or differentiated in structure and function, and so long as the differentiation follows certain prescribed lines the morphological characters of the species are preserved. We can readily conceive that, as the process of specialisation is going on, modifications or variations in groups of cells and the tissues derived from them, notwithstanding the influence of heredity, may in an individual diverge so far from that which is characteristic of the species as to assume the arrangements found in another species, or even in another order. Anatomists had indeed long recognised that variations from the customary arrangement of parts occasionally appeared, and they described such deviations from the current descriptions as irregularities.

Darwinian Theory.

The significance of the variations which arise in plants and animals had not been apprehended until a flood of light was thrown on the entire subject by the genius of Charles Darwin, who formulated the wide-reaching theory that variations could be transmitted by heredity to younger generations. In this manner he conceived new characters would arise, accumulate, and be perpetuated, which would in the course of time assume specific importance. New species might thus be evolved out of organisms originally distinct from them, and their specific characters would in turn be transmitted to their descendants. By a continuance of this process new species would multiply in many directions, until at length from one or more originally simple forms the earth would become peopled by the infinite varieties of plant and animal organisms which have in past ages inhabited, or do at present inhabit, our globe. The Darwinian theory may therefore be defined as Heredity modified and influenced by Variability. It assumes that there is an hereditary quality in the egg which, if we take the common fowl for an example, shall continue to produce similar fowls. Under conditions, of which we are ignorant, which occasion molecular changes in the cells and tissues of the developing egg, variations might arise in the first instance probably slight, but becoming intensified in successive generations, until at length the descendants would have lost the characters of the fowl and have become another species. No precise

estimate has been arrived at, and indeed one does not see how it is possible to obtain it, of the length of years which might be required to convert a variation, capable of being transmitted, into a new and definite specific character.

The circumstances which, according to the Darwinian theory, determined the perpetuation by hereditary transmission of a variety and its assumption of a specific character, depended, it was argued, on whether it possessed such properties as enabled the plant or animal in which it appeared to adapt itself more readily to its environment, *i. e.*, to the surrounding conditions. If it were to be of use the organism in so far became better adapted to hold its own in the struggle for existence with its fellows and with the forces of nature operating on it. Through the accumulation of useful characters the specific variety was perpetuated by natural selection so long as the conditions were favourable for its existence, and it survived as being the best fitted to live. In the study of the transmission of variations which may arise in the course of development, it should not be too exclusively thought that only those variations are likely to be preserved which can be of service during the life of the individual, or in the perpetuation of the species, and possibly available for the evolution of new species. It should also be kept in mind that morphological characters can be transmitted by hereditary descent, which, though doubtless of service in some bygone ancestor, are, in the new conditions of life of the species, of no physiological value. Our knowledge of the structural and functional modifications to be found in the human body, in connection with abnormalities and with tendencies or pre-disposition to diseases of various kinds, teaches us that characters which are of no use, and indeed detrimental to the individual, may be hereditarily transmitted from parents to offspring through a succession of generations.

Since the conception of the possibility of the evolution of new species from pre-existing forms took possession of the minds of naturalists, attempts have been made to trace out the lines on which it has proceeded. The first to give a systematic account of what he conceived to be the order of succession in the evolution of animals was Ernst Haeckel, of Jena, in a well-known treatise. Memoirs on special departments of the subject, too numerous to particularise, have subsequently appeared. The problem has been attacked along two different lines; the one by embryologists, of whom may be named Kowalewsky, Gegenbaur, Dohrn, Ray Lankester, Balfour, and Gaskell, who with many others have conducted careful and methodical inquiries into the stages of development of numerous forms belonging to the two great divisions of the animal kingdom. Invertebrates, as well as vertebrates, have been carefully compared with each other in the bearing of their development and structure on their affinities and descent, and the possible sequence in the evolution of the Vertebrata from the Invertebrata has been discussed. The other method pursued by palæontologists, of whom Huxley, Marsh, Cope, Osborne, and Traquair are prominent authorities, has been the study of the extinct forms preserved in the rocks, and the comparison of their structure with each other and with that of existing organisms. In the attempts to trace the line of descent the imagination has not unfrequently been called into play in constructing various conflicting hypotheses. Though from the nature of things the order of descent is, and without doubt will continue to be, ever a matter of speculation and not of demonstration, the study of the subject has been a valuable intellectual exercise and a powerful stimulant to research.

We know not as regards time when the fiat went forth, "Let there be Life, and there was Life." All we can say is that it must have been in the far-distant past, at a period so remote from the present that the mind fails to grasp the duration of the interval. Prior to its genesis our earth consisted of barren rock and desolate ocean. When

matter became endowed with Life, with the capacity of self-maintenance and of resisting external disintegrating forces, the face of nature began to undergo a momentous change. Living organisms multiplied, the land became covered with vegetation, and multitudinous varieties of plants—from the humble fungus and moss to the stately palm and oak—beautified its surface and fitted it to sustain higher kinds of living beings. Animal forms appeared, in the first instance simple in structure, to be followed by others more complex, until the mammalian type was produced. The ocean also became peopled with plant and animal organisms, from the microscopic diatom to the huge leviathan. Plants and animals acted and reacted on each other, on the atmosphere which surrounded them, and on the earth on which they dwell, the surface of which became modified in character and aspect. At last Man came into existence. His nerve-energy, in addition to regulating the processes in his economy which he possesses in common with animals, was endowed with higher powers. When translated into psychical activity it has enabled him throughout the ages to progress from the condition of a rude savage to an advanced stage of civilisation; to produce works in literature, art, and the moral sciences, which have exerted, and must continue to exert, a lasting influence on the development of his higher Being; to make discoveries in physical science; to acquire a knowledge of the structure of the earth, of the ocean in its changing aspects, of the atmosphere and the stellar universe, of the chemical composition and physical properties of matter in its various forms, and to analyse, comprehend, and subdue the forces of nature.

By the application of these discoveries to his own purposes Man has, to a large extent, overcome time and space; he has studded the ocean with steamships, girdled the earth with the electric wire, tunneled the lofty Alps, spanned the Forth with a bridge of steel, invented machines and founded industries of all kinds for the promotion of his material welfare, elaborated systems of government fitted for the management of great communities, formulated economic principles, obtained an insight into the laws of health, the causes of infective diseases, and the means of controlling and preventing them.

When we reflect that many of the most important discoveries in abstract science and in its applications have been made during the present century, and indeed since the British Association held its first meeting in the ancient capital of your county sixty-nine years ago, we may look forward with confidence to the future. Every advance in science provides a fresh platform from which a new start can be made. The human intellect is still in process of evolution. The power of application and of concentration of thought for the elucidation of scientific problems is by no means exhausted. In science is no hereditary aristocracy. The army of workers is recruited from all classes. The natural ambition of even the private in the ranks to maintain and increase the reputation of the branch of knowledge which he cultivates affords an ample guarantee that the march of science is ever onwards, and justifies us in proclaiming for the next century, as in the one fast ebbing to a close, that Great is Science, and it will prevail.

New Work on Gas Lighting.—Messrs. J. and A. Churchill announce the issue of Vol. III. of "Chemical Technology, or Chemistry in its Applications to Arts and Manufactures," edited by Charles Edward Groves, F.R.S., and William Thorp, B.Sc. This volume is on "Gas Lighting," by Charles Hunt, manager of the Birmingham Corporation Gas Works. It is expected that the work will attract the attention of all interested in the disputed subject of the advantages of gas and electric lighting. Coal of course comes under consideration.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Comptes Rendus Hebdomadaires des Seances, de l'Académie des Sciences. Vol. cxxxi., No. 7, August 13, 1900.

Action of Hydrogen on the Arsenic Sulphides.—H. Pélabon.—Hydrogen, when acting on sulphides of arsenic at temperatures above 300°, produces sulphuretted hydrogen. Inversely, sulphuretted hydrogen attacks arsenic within the same limits of temperature. The author investigates these two inverse reactions. The reagent and hydrogen were kept in sealed tubes at a given temperature of 610° for periods of five, ten, twenty, sixty, and one hundred and twenty minutes. The numbers found for the relation of the partial pressure of the hydrogen sulphide to the total pressure of mixed gases in the tube show the existence of a limit to the reaction. Using the same apparatus, the author found that sulphuretted hydrogen attacks arsenic at 610°, the reaction again being limited.

Properties of the Blue Oxide of Molybdenum.—Marcel Guichard.—In a former paper the author described the preparation of the hydrated blue oxide of molybdenum corresponding to the formula $\text{MoO}_3 \cdot 4\text{MoO}_3 \cdot 6\text{H}_2\text{O}$. A study of the properties of this blue oxide shows that it is in reality a molybdate; it only exists in a hydrated state, it being impossible to obtain anhydrous oxide of molybdenum corresponding to the blue hydrated oxide. Considering the results of the series of researches which the author has made on the various molybdenum oxides, he arrives at the following conclusion:—The number of anhydrous oxides of molybdenum, which has been stated as five by various chemists, is, in reality, two only—the anhydrous dioxides, MoO_2 , and the anhydrous trioxide, MoO_3 .

Composition of the Ashes of several Medicinal Plants.—A. B. Griffiths.

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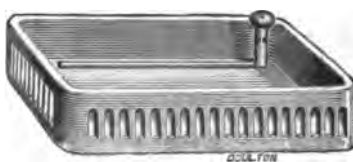
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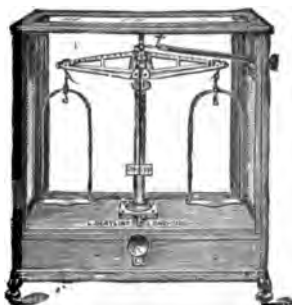
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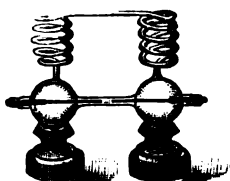
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ADDRESS TO THE CHEMICAL SECTION OF THE BRITISH ASSOCIATION.

BRADFORD, 1900.

By Professor W. H. PERKIN, Jun., Ph.D., F.R.S.,
President of the Section.

The Modern System of Teaching Practical Inorganic Chemistry and its Development.

IN choosing for the subject of my Address to-day the development of the teaching of practical inorganic chemistry I do so, not only on account of the great importance of the subject, but also because it does not appear that this matter has been brought before this Section, in the President's Address at all events, during the last few years.

In dealing generally with the subject of the teaching of chemistry as a branch of science, it may be well in the first place to consider the value of such teaching as a means of general education, and to turn our attention for a few minutes to the development of the teaching of science in schools.

There can be no doubt that there has been great progress in the teaching of science in schools during the last forty years, and this is very evident from the perusal of the essay, entitled "Education: Intellectual, Moral, and Physical," which Herbert Spencer wrote in 1859. After giving his reasons for considering the study of science of primary importance in education, Herbert Spencer continues: "While what we call civilisation could never have arisen had it not been for science, science forms scarcely an appreciable element in our so-called civilised training."

From this it is apparent that science was not taught to any appreciable extent in schools at that date, though, doubtless, in some few schools occasional lectures were given on such scientific subjects as physiology, anatomy, astronomy, and mechanics.

Herbert Spencer's pamphlet appears to have had only a very gradual effect towards the introduction of science into schemes of education. For many years chemical instruction was only given in schools at the schoolroom desk, or at the best from the lecture table, and many of the most modern of schools had no laboratories.

The first school to give any practical instruction in chemistry was apparently the City of London School, at which, in the year 1847, Mr. Hall was appointed teacher of chemistry, and there he continued to teach until 1869.* Besides the lecture theatre and a room for storing apparatus, Mr. Hall's department contained a long room, or rather passage, leading into the lecture theatre, and closed at each end with glass doors. In this room, which was fitted up as a laboratory, and used principally as a preparation room for the lectures, Mr. Hall performed experiments with the few boys who assisted him with his lectures. As accommodation was at that time strictly limited, he used to suggest simple experiments, and encourage the boys to carry them out at home, and afterwards he himself would examine the substances which they had made.

* Mr. A. T. Pollard, M.A., Head Master of the City of London School, has kindly instituted a search among the bound copies of the boys' terminal reports, and informs me that in the School form of Terminal Report a heading for Chemistry was introduced in the year 1847, the year of Mr. Hall's appointment,

From this small beginning the teaching of chemistry in the City of London School rapidly developed, and this school now possesses laboratories which compare favourably with those of any school in the country.

The Manchester Grammar School appears to have been one of the first to teach practical chemistry. In connection with this school a small laboratory was built in 1868; this was replaced by a larger one in 1872, and the present large laboratories, under the charge of Mr. Francis Jones, were opened in 1880.

Dr. Marshall Watts, who was the first science master in this school, taught practical chemistry along with the theoretical work from the commencement in 1868.

As laboratories were gradually multiplied, it might be supposed that boys were given the opportunity to carry out experiments which had a close connection with their lecture-room courses. But the programme of laboratory work which became all but universal was the preparation of a few gases, followed by the practice of qualitative analysis. The course adopted seems to have been largely built up on the best books of practical chemistry in use in the colleges at that time; but it was also, no doubt, largely influenced by the requirements of the syllabus of the Science and Art Department, which contained a scheme for teaching practical chemistry.* Even down to quite recent times it was in many schools still not considered essential that boys should have practical instruction in connection with lectures in chemistry.

A Report issued in 1897 by a special Committee appointed by the Technical Education Board of the London County Council, adduces evidence of this from twenty-five secondary schools in London, in which there were 3960 boys learning chemistry. Of these 1698 boys, or 43 per cent, did no practical work whatever; 955 boys, or 24 per cent, did practical work, consisting of a certain amount of preparation of gases, together with qualitative analysis; but of these latter 743, or 77 per cent, had not reached the study of the metals in their theoretical work, so that their testing work can have been of little educational value. It was also found that in the case of 655, or 68 per cent of the total number of boys taking practical work, the first introduction to practical chemistry was through qualitative analysis.

But some years before this Report was issued, a movement had begun which was destined to have a far-reaching effect. A Report "on the best means for promoting Scientific Education in Schools" having been presented to the Dundee Meeting of this Association in 1867, and published in 1868, a Committee of the British Association was appointed in 1887 "for the purpose of inquiring and reporting upon the present methods of teaching chemistry." The well-known Report which this Committee presented to the Newcastle Meeting in 1889 insisted that it was worth while to teach chemistry in schools, not so much for the usefulness of the information imparted, as for the special mental discipline it afforded if the scientific method of investigating nature were employed. It was argued that "learners should be put in the attitude of discoverers, and led to make observations, experiments, and inferences for themselves." And since there can be little progress without measurement, it was pointed out that the experimental work would necessarily be largely of a quantitative character.

Professor H. E. Armstrong, in a paper read at a conference at the Health Exhibition five years before this, had foreshadowed much that was in this Report. He also drew up a detailed scheme for "a course of elementary instruction in physical science," which was included in the Report of the Committee, and it cannot be doubted that this scheme and the labours of the Committee have had a very marked influence on the development of the teaching of practical chemistry in schools. That this in-

* I find, on inquiry, that examinations in the Advanced Stage and Honours of Practical Chemistry were first held by the Science and Art Department in 1878, the practical examination being extended to the Elementary Stage in 1882.

fluence has been great will be admitted when it is understood that schemes based on the recommendation of the Committee are now included in the codes for both Elementary Day Schools and Evening Continuation Schools. The recent syllabuses for elementary and advanced courses issued by the Incorporated Association of Headmasters, and by the Oxford and Cambridge local boards and others, are evidently directly inspired by the ideas set forth by the Committee.

The Department of Science and Art has also adopted some of the suggestions of the Committee, and a revised syllabus was issued by the Department in 1895, in which qualitative analysis is replaced by quantitative experiments of a simple form, and by other exercises so framed "as to prevent answers being given by students who have obtained their information from books or oral instruction." This was a very considerable advance, but it must be admitted that there is nothing in the syllabus which encourages, or even suggests, placing the learners in the attitude of discoverers, and this, in the opinion of the Committee of this Association, is vital if the teaching is to have educational value.

Many criticisms have been passed upon the 1889 Report. It has been said that life is much too short to allow of each individual advancing from the known to the unknown, according to scientific methods, and that even were this not so too severe a tax is made upon the powers of boys and girls. In answer to the second point it will be conceded that while it is doubtless futile to try to teach chemistry to young children, on the other hand experience has abundantly shown that the average schoolboy of fourteen or fifteen can, with much success, investigate such problems as were studied in the researches of Black and Scheele, of Priestley and Cavendish, and Lavoisier, and it is quite remarkable with what interest such young students carry out this class of work.

It may be well to quote the words which Sir Michael Foster used in this connection in his admirable Presidential Address to this Association in 1899. He said:—"The learner may be led to old truths, even the oldest, in more ways than one. He may be brought abruptly to a truth in its finished form, coming straight to it like a thief climbing over a wall; and the hurry and press of modern life tempt many to adopt this quicker way. Or he may be more slowly guided along the path by which the truth was reached by him who first laid hold of it. It is by this latter way of learning the truth, and by this alone, that the learner may hope to catch something at least of the spirit of the scientific inquirer."

I believe that in the determination of a suitable school course in experimental science this principle of historical development is a very valuable guide, although it is not laid down in the 1889 Report of the British Association.

The application of this principle will lead to the study of the solvent action of water, of crystallisation, and of the separation of mixtures of solids before the investigation of the composition of water, and also before the investigation of the phenomena of combustion. It will lead to the investigation of hydrochloric acid before chlorine, and especially to the postponement of atomic and molecular theories, chemical equations, and the laws of chemical combination, until the student has really sufficient knowledge to understand how these theories came to be necessary.

There can be no doubt that this new system of teaching chemistry in schools has been most successful. Teachers are delighted with the results which have already been obtained, and those whom I have had the opportunity of consulting, directly and indirectly, cannot speak too highly of their satisfaction at the disappearance of the old system of qualitative analysis, and the institution of the new order of things. Especially I may mention in this connection the excellent work which is being carried on under the supervision of Dr. Bevan Lean at the Friends' School in Ackworth, where the boys have attained re-

sults which are far in advance of anything which would have been thought possible a few years since.

It is, of course, obvious that if a schoolboy is made to take the attitude of a discoverer his progress may appear to be slow. But does this matter? Most boys will not become professional chemists; but if while at school a boy learns how to learn, and how to "make knowledge" (cf. Professor J. G. Macgregor in *Nature*, September, 1899) by working out for himself a few problems, a habit of mind will be formed which will enable him in future years to look in a scientific spirit at any new problems which may face him. When school days are past the details of the preparation of hydrogen may have been forgotten; but if it was really understood at the time that it could not be decided at once whether the gas was derived from the acid or from the metal, or from the water, or in part from the one and in part from the other, an attitude of scepticism and of suspended judgment will have been formed, which will continue to guard from error.

In the new system of teaching chemistry in schools much attention must necessarily be given to weights and measurements; indeed, the work must be largely of a quantitative kind, and it is in this connection that an important note of warning has been sounded by several teachers (cf. H. Piéron in *The School World*, November, 1899; Bevan Lean, *ibid.*, February, 1890). They consider, very rightly, that it is important to point out clearly to the scholar that science does not consist of measurement, but that measurement is only a tool in the hand of the inquirer, and that when once sufficient skill has been developed in its use it should be employed only with a distinct object. Measurements should, in fact, be made only in reference to some actual problem which appears to be really worth solving, not in the accumulation of aimless details.

And, of course, all research carried out must be genuine and not sham, and all assumption of the "obvious" must be most carefully guarded against. But the young scholar must, at the same time, not forget that although the scientific method is necessary to enable him to arrive at a result, in real life it is the answer to the problem which is of the most importance (cf. Mrs. Bryant, *Special Reports on Educational Subjects*, vol. ii., p. 113).

Although, then, there has been so much discussion, during the last ten years, on the subject of teaching chemistry in schools, and such steady progress has been made towards devising a really satisfactory system of teaching the subject to young boys and girls, it is certainly very remarkable that practically nothing has been said or written bearing on the training which a student, who wishes to become a chemist, is to undertake at the close of his school days at the college or university in which his education is continued.

One of the most remarkable points, to my mind, in connection with the teaching of chemistry, is the fact that although the science has been advancing year by year with such unexampled rapidity, the course of training which the student goes through during his first two years at most colleges is still practically the same as it was thirty or forty years ago. Then, as now, after preparing a few of the principal gases, the student devotes the bulk of his first year to qualitative analysis in the dry and wet way, and his second year to quantitative analysis, and, although the methods employed in teaching the latter may possibly have undergone some slight modification, there is certainly no great difference between the routine of simple salt and mixture, followed by quantitative analysis practised at the present day, and that which was in vogue in the days of our fathers and grandfathers.

Since, then, the present system has held the field for so long, not only in this country but also on the Continent, it is worth while considering whether it affords the best training which a student who wishes to become a chemist can undergo in the short time during which he can attend at a college or university. In considering this matter I was led in the first place to carefully examine old books

and other records, with the object of finding out how the present system originated, and I think that valuable and interesting information bearing on the subject may be obtained from a very brief sketch of the rise and development of the present system of teaching chemistry, and especially in so far as it bears on the inclusion of qualitative analysis. Unfortunately, it is not so easy to gain a good historical acquaintance with the matter as I at first imagined would be the case, and this is due in a large measure to the fact that so few of the laboratories which took an active part in the development of the present system of chemical training have left any record of the methods which they employed. In this connection I may perhaps be allowed to suggest that it would be a valuable help to the future historian if all prominent teachers of chemistry would leave behind them a brief record of the system of teaching adopted in their laboratories, showing the changes which they had instituted, the object of these changes, and the results which followed their adoption.

There is no doubt that the progress of practical chemistry went largely hand in hand with the progress of theoretical chemistry, for as the latter gradually developed, so the necessity for the determination of the composition, first of the best known, and then of the rarer minerals and other substances, became more and more marked.

The analytical examination of substances in the dry way was employed in very early times in connection with metallurgical operations, and especially in the determination of the presence of valuable constituents in samples of minerals. Cupellation was used by the Greeks in the separation of gold and silver from their ores and in the purification of these metals. Geber knew that the addition of nitre to the ore facilitated the separation of gold and silver, and subsequently Glauber (1604-1668) called attention to the fact that many commoner metals could easily be separated from their ores with the aid of nitre.

But it was not till the eighteenth century that any marked progress was made in analysis in the dry way, and the progress which then became rapid was undoubtedly due to the discovery of the blowpipe, and to the introduction of its use into analytical operations. The blowpipe is mentioned for the first time in 1660, in the *Transactions of the Accademia del Cimento* of Florence, but the first to recommend its use in chemical operations was Johann Andreas Cramer in 1739. The progress of blowpipe analysis was largely due to Gahn (1745-1818), who spent much time in perfecting its use in the examination of minerals, and it was he who first used platinum wire and cobalt solution in connection with blowpipe analysis. The methods employed by Gahn were further developed by his friend Berzelius (1779-1848), who gave much attention to the matter, and who with great skill and patience gradually worked out a complete scheme of blowpipe analysis, and published it in a pamphlet entitled "*Ueber die Anwendung des Löthrohrs*," which appeared in 1820. After the publication of this work blowpipe analysis rapidly came into general use in England, France, and Germany, and the scheme devised by Berzelius is essentially that employed at the present day.

Indeed, the only notable additions to the methods of analysis in the dry way since the time of Berzelius are the development of flame reactions, which Bunsen worked out with such characteristic skill and ingenuity, and the introduction of the spectroscope.

The necessity for some process other than that of analysis in the dry way seems, in the first instance, to have arisen in quite early times in connection with the examination of drugs, not only on account of the necessity for discovering their constituents, but also as a means of determining whether they were adulterated. In such cases analysis in the dry way was obviously unsuitable, and experience soon showed that the only way to arrive at the desired result was to treat the substance under

examination with aqueous solutions of definite substances, the first reagent apparently being a decoction of gallnuts, which is described by Pliny as being employed in detecting adulteration with green vitriol.

The progress made in connection with wet analysis was, however, exceedingly slow, largely owing to the lack of reagents; but as these were gradually discovered wet analysis rapidly developed, especially in the hands of Tachenius, Scheele, Boyle, Hoffman, Margraf, and Bergmann. Boyle (1626-1691) especially had an extensive knowledge of reagents and their application; and, indeed, it was Boyle who first introduced the word "analysis" for those operations by which substances may be recognised in the presence of one another. Boyle knew how to test for silver with hydrochloric acid, for calcium salts with sulphuric acid, and for copper by the blue solution produced by ammonia.

Margraf (1709-1782) introduced prussiate of potash for the detection of iron, and Bergmann (1735-1784) not only introduced new reagents and new methods for decomposing minerals and refractory substances, such as fusion with potash, digestion with nitric acid or hydrochloric acid, but he also was the first to suggest the application of tests in a systematic way, and, indeed, the method of analysis which he developed is on much the same lines as that in use at the present day. He paid special attention to the qualitative analysis of minerals, and gave careful instructions for the analysis of gold, platinum, silver, lead, copper, zinc, and other ores. The work of Scheele (1742-1786) had indirectly a great influence on qualitative analysis, as, although he did not give a general systematic method of procedure in the analysis of substances of unknown composition, yet the methods which he employed in the examination of new substances were so original and exact as to remain models of how qualitative analysis should be conducted.

Great strides in analytical chemistry in the wet way were made through the work of Berzelius, who, by the discovery of new methods, such as the decomposition of silicates by hydrofluoric acid and the introduction of new tests, greatly advanced the art. He paid special attention to perfecting the methods of analysis of mineral waters, and these researches as well as his work on ores, and particularly his investigation of platinum ores, stamp Berzelius as one of the great pioneers in qualitative and quantitative analytical chemistry.

By the labours of the great experimenters whom I have mentioned qualitative analysis gradually acquired the familiar appearance of to-day, and many books were written with the object of arranging the mass of information which had accumulated, and of thus rendering it available for the student in his efforts to investigate the composition of new minerals and other substances. Among these books may be mentioned the "*Handbuch der Analytischen Chemie*," by H. Rose, and especially the well-known analytical text-books of Fresenius, which have had an extraordinarily wide circulation and passed through many editions.

The work of the great pioneers in analytical chemistry was work done often under circumstances of great difficulty, as before the end of the seventeenth century there were no public institutions of any sort in which a practical knowledge of chemistry could be acquired. Lectures were, of course, given from very early times, but it was not until the time of Guillaume François Rouelle (1703-1770), at the beginning of the eighteenth century, that lectures began to be illustrated by experiments. Rouelle, who was very active as a teacher, numbered among his pupils many men of eminence, such as Lavoisier and Proust, and it was largely owing to his influence that France took such a lead in practical teaching. In Germany progress was much slower, and in our country the introduction of lectures illustrated by experiments seems to have been mainly due to Davy.

When it is considered how slowly experimental work came to be recognised as a means of illustration and

education, even in connection with lectures, it is not surprising that in early times practical teaching in laboratories should have been thought quite unnecessary.

The few laboratories which existed in the sixteenth century were built mainly for the practice of alchemy by the reigning princes of the time, and, indeed up to the beginning of the nineteenth century, the private laboratories of the great masters were the only schools in which a favoured few might study, but which were not open to the public. Thus we find that Berzelius received in his laboratory a limited number of students who worked mostly at research: these were not usually young men, and his school cannot thus be considered as a teaching institution in the ordinary sense of the word.

The earliest laboratory open for general instruction in Great Britain was that of Thomas Thomson, who, after graduating in Edinburgh in 1799, began lecturing in that city in 1800, and opened a laboratory for the practical instruction of his pupils. Thomson was appointed Lecturer in Chemistry in Glasgow University in 1807, and Regius Professor in 1818, and in Glasgow he also opened a general laboratory.

The first really great advance in laboratory teaching is due to Liebig, who, after working for some years in Paris under Gay-Lussac, was appointed in 1824 to be Professor of Chemistry in Giessen. Liebig was strongly impressed with the necessity for public institutions where any student could study chemistry, and to him fell the honour of founding the world-famed Giessen Laboratory, the first public institution in Germany which brought practical chemistry within the reach of all students.

Giessen rapidly became the centre of chemical interest in Germany, and students flocked to the laboratory in such numbers as to necessitate the development of a systematic course of practical chemistry, and in this way a scheme of teaching was devised which, as we shall see later, has served as the foundation for the system of practical chemistry in use at the present day.

When the success of this laboratory had been clearly established many other towns discovered the necessity for similar institutions, and in a comparatively short time every university in Germany possessed a chemical laboratory. The teaching of practical chemistry in other countries was, however, of very slow growth; in France, for example, Wurtz in 1869 drew attention to the fact that there was at that time only one laboratory which could compare with the German laboratories, namely, that of the Ecole Normale Supérieure.

In this country the provision of suitable laboratories for the study of chemistry seems to date from the year 1845, when the College of Chemistry was founded in London, an institution which under A. W. Hofmann's guidance rapidly rose to such a prominent position.

In 1851 Frankland was appointed to the chair of chemistry in the new college founded in Manchester by the trustees of John Owens, and here he equipped a laboratory for the teaching of practical chemistry. Under Sir Henry Roscoe this laboratory soon became too small for the growing number of chemical students, a defect which was removed when the new buildings of the college were opened in 1873. In 1849 Alexander Williamson was appointed Professor of Practical Chemistry at University College, London, where he introduced the practical methods of Liebig.

Following these examples, the older universities gradually came to see the necessity for providing accommodation for the practical teaching of chemistry, with the result that well-equipped laboratories have been erected in all the centres of learning in this country.

Since Liebig, by the establishment of the Giessen Laboratory, must be looked upon as the pioneer in the development of practical laboratory teaching, it will be interesting to endeavour to obtain some idea of the methods which he used in the training of the students who attended his laboratory in Giessen. From small beginnings he gradually introduced a systematic course of practical

chemistry, and a careful comparison shows that this was similar in many ways to that in use at the present day. The student at Giessen, after preparing the more important gases, was carefully trained in qualitative and quantitative analysis; he was then required to make a large number of preparations, after which he engaged in original research.

Although there is, as far as I have been able to ascertain, no printed record of the nature of the quantitative work and the preparations which Liebig required from his students, the course of qualitative analysis is easily followed, owing to the existence of a most interesting book published for the use of the Giessen students.

In 1846, at Liebig's request, Henry Will, Ph.D., Extraordinary Professor of Chemistry in the University of Giessen, wrote a small book, for use at Giessen, called "Giessen Outlines of Analysis," which shows clearly the kind of instruction given in that laboratory at the time in so far as qualitative analysis is concerned. This book, which contains a preface by Liebig, is particularly interesting on account of the fact that it is evidently the first introduction to Analysis intended for the training of elementary students which was ever published. In the preface Liebig writes:—"The want of an introduction to chemical analysis adapted for the use of a laboratory has given rise to the present work, which contains an accurate description of the course I have followed in my laboratory with great advantage for twenty-five years. It has been prepared at my request by Professor Will, who has been my assistant during a great part of this period."

This book undoubtedly had a considerable circulation, and was used in most of the laboratories which were in existence at that time, and thus we find, for example, that the English translation, which Liebig "hopes and believes will be acceptable to the English public" was the book used by Hofmann for his students at the College of Chemistry. In this book the metals are first divided into groups much in the same way as is done now; each group is then separately dealt with, the principal characteristics of the metals of the group are noted, and their reactions studied. Those tests which are useful in the detection of each metal are particularly emphasised, and the reasons given for selecting certain of them as of special value for the purposes of separating one metal from another.

Throughout this section of the book there are frequent discussions as to the possible methods of the separation, not only of the metals of one group, but of those belonging to different groups; and the whole subject is treated in a manner which shows clearly that Liebig's great object was to make the student think for himself. After studying in a similar manner the behaviour of the principal acids with reagents, the student is introduced to a course of qualitative analysis comprising, 1, preliminary examination of solids; 2, qualitative analysis of the substance in solution.

Both sections are evidently written with the object, not only of constructing a system of qualitative analysis, but more particularly of clearly leading the student to argue out for himself the methods of separation which he will ultimately adopt. The book concludes with a few tables, which differ considerably in design from those in use at the present day, and which are so meagre that the student could not possibly have used them mechanically.

The system introduced in this book, no doubt owing to the excellent results obtained by its use, was rapidly recognised as the standard method of teaching analysis in most of the institutions existing at that time. Soon the course began to be further developed, book after book was published on the subject, and gradually the teaching of qualitative analysis assumed the shape and form with which we are all so well acquainted. But the present-day book on qualitative analysis differs widely from "Giessen Outlines" in this respect, that whereas in the latter the tables introduced are mere indications of the methods of separation to be employed, and are of such a nature that the student who did not think for himself must have been constantly in difficulties, in the book of the present day

these tables have been worked out to the minutest detail. Every contingency is provided for; nothing is left to the originality of the student; and that which, no doubt, was once an excellent course has now become so hopelessly mechanical as to make it doubtful whether it retains anything of its former educational value.

The question which I now wish to consider more particularly is whether the system of training chemists which is at present adopted, with little variation, in our colleges and universities, is a really satisfactory one, and whether it supplies the student with the kind of knowledge which will be of the most value to him in his future career.

Those who study chemistry may be roughly divided as to their future careers into two groups—those who become teachers and those who become technical chemists. Now, whether the student takes up either the one or the other career, I think that it is clear that the objects to be aimed at in training him are to give him a sound knowledge of his subject, and especially to so arrange his studies as to bring out in every possible way his capacity for original thought.

A teacher who has no originality will hardly be successful, even though he may possess a very wide knowledge of what has already been done in the past. He will have little enthusiasm for his subject, and will continue to teach on the lines laid down by the text-books of the day, without himself materially improving the existing methods, and, above all, he will be unable, and will have no desire, to add to our store of knowledge by original investigation.

It is in the power of almost every teacher to do some research work, and it seems probable that the reason why more is not done by teachers is because the importance of research work was not sufficiently insisted on, and their original faculty was not sufficiently trained, at the schools and colleges where they received their education.

And these remarks apply with equal force to the student who subsequently becomes a technical chemist.

In the chemical works of to-day sound knowledge is essential, but originality is an even more important matter. A technical chemist without originality can scarcely rise to a responsible position in a large works, whereas a chemist who is capable of constantly improving the processes in operation, and of adding new methods to those in use, becomes so valuable that he can command his own terms.

Now, this being so, I think it is extraordinary that so many of the students who go through the prescribed course of training—say for the Bachelor of Science degree—not only show no originality themselves, but seem also to have no desire at the conclusion of their studies to engage in original investigation under the supervision of the teacher. That this is so is certainly my experience as a teacher examiner, and I feel sure that many other teachers will endorse this view of the case.

If we inquire into the reason for this deficiency in originality we shall, I think, be forced to conclude that it is in a large measure due to the conditions of study and the nature of the courses through which the student is obliged to pass.

A well-devised system of quantitative analysis is undoubtedly valuable in teaching the student accurate manipulation, but it has always seemed to me that the long course of qualitative analysis, which is usually considered necessary, and which generally precedes the quantitative work, is not the most satisfactory training for a student.

There can be no doubt that to many students qualitative analysis is little more than a mechanical exercise; the tables of separation are learnt by heart, and every substance is treated in precisely the same manner; such a course is surely not calculated to develop any original faculty which the student may possess. Then, again, when the student passes on to quantitative analysis, he receives elaborate instructions as to the little details he must observe in order to get an accurate result; and even after he has become familiar with the simpler determin-

ations he rarely attempts, and indeed has no time to attempt, anything of the nature of an original investigation in qualitative or quantitative analysis. It indeed sometimes happens that a student at the end of his second year has never prepared a pure substance, and is often utterly ignorant of the methods employed in the separation of substances by crystallisation; he has never conducted a distillation, and has no idea how to investigate the nature and amounts of substances formed in chemical reactions; practically all his time has been taken up with analysis. That this is not the way to teach chemistry was certainly the opinion of Liebig, and in support of this I quote a paragraph bearing on the subject, which occurs in a very interesting book on "Justus von Liebig: his Life and Work," written by W. A. Shenstone (pp. 175, 176).

"In his practical teaching Liebig laid great stress on the producing of chemical preparations; on the students preparing, that is to say, pure substances in good quantity from crude materials. The importance of this was, even in Liebig's time, often overlooked; and it was, he tells us, more common to find a man who could make a good analysis than to find one who could produce a pure preparation in the most judicious way."

"There is no better way of making one's self acquainted with the properties of a substance than by first producing it from the raw material, then converting it into its compounds, and so becoming acquainted with them. By the study of ordinary analysis one does not learn how to use the important methods of crystallisation, fractional distillation, nor acquire any considerable experience in the proper use of solvents. In short, one does not, as Liebig said, become a chemist."

One reason why the present system of training chemists has persisted so long is, no doubt, because it is a very convenient system; it is easily taught, does not require expensive apparatus, and, above all, it lends itself admirably for the purpose of competitive examination.

The system of examination which has been developed during the last twenty years has done much harm, and is a source of great difficulty to any conscientious teacher who is possessed of originality, and is desirous, particularly in special cases, of leaving the beaten track.

In our colleges and universities most of the students work for some definite examination—frequently for the Bachelor of Science degree—either at their own University or at the University of London.

For such degrees a perfectly definite course is prescribed and must be followed, because the questions which the candidate will have to answer at his examination are based on a syllabus, which is either published or is known by precedent to be required. The course which the teacher is obliged to teach is thus placed beyond his individual power of alteration, except in minor details, and originality in the teacher is thereby discouraged; he knows that all students must face the same examination, and he must urge the backward man through exactly the same course as his more talented neighbour.

In almost all examinations salts or mixtures of salts are given for qualitative analysis. "Determine the constituents of the simple salt A and of the mixture B" is a favourite examination formula; and as some practical work of this sort is sure to be set, the teacher knows that he must contrive to get one and all of his students into a condition to enable them to answer such questions.

If, then, one considers the great amount of work which is required from the present-day student, it is not surprising that every aid to rapid preparation for examination should be accepted with delight by the teacher; and thus it comes about that tables are elaborated in every detail, not only for qualitative analysis in inorganic chemistry, but, what is far worse, for the detection of some arbitrary selection of organic substances which may be set in the syllabus for examination. I question whether any really competent teacher will be found to recommend this system as one of educational value, or calculated to

bring out and train the faculty of original thought in students.

If, then, the present system is so unsatisfactory, it will na ually be asked, How are students to be trained, and how are they to be examined, so as to find out the extent of the knowledge of their subject which they have acquired?

In dealing with the first part of the question—that is, the training best suited to chemists—I can, of course, only give my own views on the subject—views which, no doubt, may differ much from those of many of the teachers present at this meeting. The objects to be attained are, in my opinion, to give the student a sufficient knowledge of the broad facts of chemistry, and at the same time so to arrange his practical work in particular as to always have in view the training of his faculty of original thought.

I think it will be conceded that any student, if he is to make his mark in chemistry by original work, must ultimately specialise in some branch of the subject. It may be possible for some great minds to do valuable original work in more than one branch of chemistry, but these are the exceptions; and as time goes on, and the mass of facts accumulates, this will become more and more impossible. Now a student at the commencement of his career rarely knows which branch of the subject will fascinate him most, and I think, therefore, that it is necessary, in the first place, to do all that is possible to give him a thorough grounding in all branches of the subject. In my opinion the student is taken over too much ground in the lecture courses of the present day; in inorganic chemistry, for example, the study of the rare metals and their reactions might be dispensed with, as well as many of the more difficult chapters of physical chemistry, and in organic chemistry such complicated problems as the constitutions of uric acid and the members of the camphor and terpene series, &c., might well be left out. As matters stand now, instruction must be given on these subjects simply because questions bearing on them will probably be asked at the examination.

And here perhaps I might make a confession, in which I do not ask my fellow teachers to join me. My name is often attached to chemistry papers which I should be sorry to have to answer, and it seems to me the standard of examination papers, and especially of honours examination papers, is far too high. Should we demand a pitch of knowledge which our own experience tells us cannot be maintained for long?

In dealing with the question of teaching practical chemistry it may be hoped, in the first place, that in the near future a sound training will be given in elementary science in most schools, very much on the lines which I mentioned in the first part of this Address. The student will then be in a fit state to undergo a thoroughly satisfactory course of training in inorganic chemistry during his first two years at college. Without wishing in any way to map out a definite course, I may be allowed to suggest that instead of much of the usual qualitative and quantitative analysis, practical exercises, similar to the following, will be found to be of much greater educational value.

(1). The careful experimental demonstration of the fundamental laws of chemistry and physical chemistry.

(2). The preparation of a series of compounds of the more important metals, either from their more common ores or from the metals themselves. With the aid of the compounds thus prepared the reactions of the different metals might be studied, and the similarities and differences between the different metals then carefully noted.

(3). A course in which the student should investigate in certain selected cases: (a) the conditions under which action takes place; (b) the nature of the products formed; (c) the yield obtained. If he were then to proceed to prepare each product in a state of purity, he would be doing a series of exercises of the highest educational value.

(4). The determination of the combining weights of some of the more important metals. This is, in most cases, comparatively simple, as the determination of the combining weights of selected metals can be very accurately carried out by measuring the hydrogen evolved when an acid acts upon them.

Many other exercises of a similar nature will readily suggest themselves, and in arranging the course every effort should be made to induce the student to consult original papers, and to avoid, as far as possible, any tendency to mere mechanical work.

The exact nature of such a course must, however, necessarily be left very much in the hands of the teacher, and the details will no doubt require much consideration; but I feel sure that a course of practical inorganic chemistry could be constructed which, while teaching all the important facts which it is necessary for the student to know, will, at the same time, constantly tend to develop his faculty of original thought.

Supposing such a course were adopted (and the experiment is well worth trying), there still remains the problem of how the student who has had this kind of training is to be examined.

With regard to his theoretical work there would be no difficulty, as the examination could be conducted on much the same lines as at the present time. In the case of the practical examination, I have long felt that the only satisfactory method of arriving at the value of a student's practical knowledge is by the inspection of the work which he has done during the whole of his course of study, and not by depending on the results of one or two days' set examination. I think that most examiners will agree with me that the present system of examination in practical chemistry is highly unsatisfactory. This is perhaps not so apparent in the case of the qualitative analysis of the usual simple salt or mixture; but when the student has to do a quantitative exercise, or when a problem is set, the results sent in are frequently no indication of the value of the student's practical work. Leaving out of the question the possibility of the student being in indifferent health during the short period of the practical examination, it not infrequently happens that he, in his excitement, has the misfortune to upset a beaker when his quantitative determination is nearly finished, and as a result he loses far more marks than he should do for so simple an accident.

Again, in attacking a problem he has usually only time to try one method of solution, and if this does not yield satisfactory results he again loses marks, whereas in the ordinary course of his practical work, if he were to find that the first method was faulty he would try other methods, until he ultimately arrived at the desired result.

It is difficult to see why such an unsatisfactory system as this might not be replaced by one of inspection, which I think could easily be so arranged as to work well.

A student taking, say, a three years' course for the degree of Bachelor of Science might be required to keep very careful notes of all the practical work which he does during this course, and in order to avoid fraud his notebook could from time to time be initialed by the professor or demonstrator in charge of the laboratory. An inspection of these notebooks could then be made at suitable times by the examiners for the degree, by which means a very good idea would be obtained of the scope of the work which the student had been engaged in, and if thought necessary a few questions could easily be asked in regard to the work so presented. Should the examiners wish to further test the candidate by giving him an examination, I submit that it would be much better to set him some exercise of the nature of a simple original investigation, and to allow him two or three weeks to carry this out, than to depend on the hurried work of two or three days.

The object which I had in view in writing this Address was to call attention to the fact that our present system

of training in chemistry does not appear to develop in the student the power of conducting original research, and at the same time to endeavour to suggest some means by which a more satisfactory state of things might be brought about. I have not been able, within the limits of this Address, to consider the conditions of study during the third year of the student's career at college, or to discuss the increasing necessity for extending that course, and insisting on the student carrying out an adequate original investigation before granting him a degree, but I hope on some future occasion to have the opportunity of returning to this very important part of the subject. If any of the suggestions I have made should prove to be of practical value, and should lead to the production of more original research by our students, I shall feel that a useful purpose has been served by bringing this matter before this Section. In concluding, I wish to thank Professor H. B. Dixon, Professor F. S. Kipping, and others, for many valuable suggestions, and my thanks are especially due to Dr. Bevan Lean for much information which he gave me in connection with that part of this Address which deals with the teaching of chemistry in schools.

SOME NEW SPECTRA OF RARE EARTHS.

By EUG. DEMARCAV.

TERBIA, one of the rare earths whose existence has been certain for a long time, owing to the existence of its coloured peroxide, is extremely ill-defined, because no spectra of terbia is known, nor even the true colour of the pure peroxide.

M. Lecoq de Boisbaudran showed that this substance, to which a pale colour had been attributed, was in reality very dark. However, even then it is not yet pure. I was able, indeed, to obtain from a specimen which was almost black, a spark spectrum in which several metals were present in apparently equal quantities. We can predict, however, without too great risk of error, that pure terbia peroxide will be as dark in colour as praseodymium peroxide, and that the simple yellow earths are contained only in very small quantities.

In the spectra of these various terbias I noticed several lines which seemed to belong to terbium. They were strongly marked in the spectrum of M. Lecoq de Boisbaudran's brown terbia, together with lines belonging to various other elements. I obtained these terbia lines, together with the gadolinium lines, in the most soluble product of the magnesium nitrate of gadolinium which was already almost pure.

This very soluble product gives a brown oxide and a trace of the absorption band $\lambda=4877$, which M. Lecoq has attributed to Z_4 . I give the eight strongest lines belonging to this spectrum.

λ	Intensity.	λ	Intensity.
3704.3	10	3561.7	12
3703.2	11	3540.2	11
3676.7	12	3523.4	10
3568.4	10	3508.5	12

The maximum intensity being 16, minimum 1,

I provisionally have called the element having the above spectrum Γ . In the spectra of products rich in terbia, but approaching holmium, a second series of lines is apparent, brought out by certain fractionations in the less-coloured earth.

These correspond to a particular element in the neighbourhood of holmium, abundant in the dysprosium earths. Perhaps it is identical with Lecoq's earth Z_7 , identified by a band spectrum in the yellow portion of the spectrum. I shall call this element Δ , leaving the question as to its identity with Z_7 open.

The most prominent lines of this spectrum are:—

λ	Intensity.	λ	Intensity.
4212.6	11	3945.0	10
4195.5	6	3595.0	6
4187.3	9	3550.0	5
3978.6	6	3531.3	11

In the spectrum of the yttria earths, which are still more soluble and are intermediate between holmium and erbium, I notice two lines which are not due to either holmium or erbium. These two lines, which can be obtained very strong, have wave-lengths 3967.9 and 3930.9, and appear to belong to a particular element, which I will call Ω .

Finally, in the very slightly basic earths intermediate between erbium and ytterbium, several strong lines are seen, of which two (wave-lengths 4008.2 and 3906.5) do not seem to belong to thulium.

These strong lines may belong to $\Sigma-Z_4$. I am calling the elements to which these lines belong Θ .

I have also noticed, in addition to the various spectra mentioned above, a number of other lines due to elements already recognised by their absorption spectra. These spectra are not so marked as the others, having a much less bright spark.

As with the others, I have only obtained them as a mixture. The greater number of their lines and their faintness render the elements to which they belong still uncertain.—*Comptes Rendus*, cxxxi., No. 6.

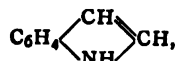
SYNTHETIC INDIGO.*

By EDWARD S. JOHNSON.

(Continued from p. 105).

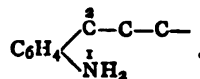
II. THE CONSTITUTION OF INDIGO.

THE leading motive of the classic investigations of Baeyer in the indigo series was the desire to establish "the place of each atom in the indigo molecule experimentally" (Baeyer, "Ueber d. Verb. Indigoreihe," *Ber. Deut. Chem. Gesell.*, xvi., 2188) and the relation of all bodies of the group to indol,—

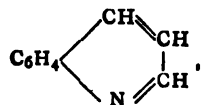


as generatrix (*Ber. Deut. Chem. Gesell.*, xv., 775).

It was by no mere chance that the formation of indigo from *o*-nitrocinnamic acid was discovered, but by a deliberately formed conclusion drawn from the facts observed in studying the linking of the nitrogen atom of an amido-group of a benzene ring, in ortho-position to a chain of two or more carbon atoms, thus—



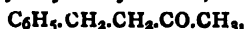
It was thereby and in the synthesis of bodies of the indigo group† found that the nitrogen of the amido-group unites with the second or third carbon atom of the chain, but not, it seems, with those more distant. The formation of such rings is not dependent upon the presence of the carboxyl-group. Thus a substance resembling quinoline,—



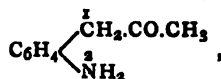
* *Proceedings of Engineers' Society of Western Pennsylvania*, xvi., No. 3.

† Throughout, the original discussion of the subject, as found in the literature to which reference has been or will be made, is here or will be in other similar instances closely followed.

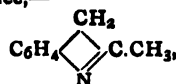
is formed when the third carbon atom is present in ketone-form as in phenylethylmethyl ketone,—



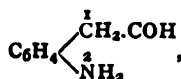
and it may be said in advance, with great probability of correctness, that whenever the second or third carbon atom is present as an alcohol, aldehyde, or ketone group, an inner anhydride will result, which belongs either to the indol or quinoline series. It was then further observed that the methylketone of *o*-amidophenylacetic acid,—



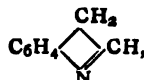
produces a substance,—



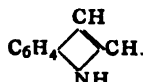
of the composition of a methylindol. If a ketone shows this reaction, it is to be expected that the corresponding aldehyde,—



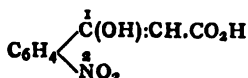
would give a body,



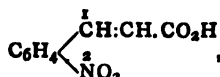
isomeric with indol,—



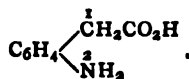
For this purpose, exactly as in the unnitrated body, it would be necessary to prepare the aldehyde by means of *o*-nitrophenyloxacrylic acid,—



This led then to the conviction that in *o*-nitrocinnamic acid,—



a better starting-point for the preparation of indigo, isatin, and indol would then be found in *o*-amidophenylacetic acid,—

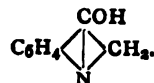


despite the loss suffered by the splitting off of carbonic anhydride (Bayer, "Ueber d. Bezieh. d. Zimmtsäure zu d. Indogruppe," *Ber. Deut. Chem. Gesell.*, xiii., 2254).

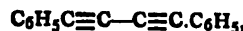
A series of most remarkable substances was developed—*o*-nitrophenylpropionic acid, isatogenic acid, indoxyllic acid, indoxyl and derivatives, indoxanthinic acid, dinitrodiphenyldiacetylene, di-isatogen—and gave up under the persistent and skilful inquisition and acute perception of their discoverer the secret of the constitution of indigo.

The way through *o*-nitrophenylpropionic, isatogenic, and indoxyllic acids to indoxyl gave important insight into the question. Indoxyl warmed with *o*-nitrophenylpropionic acid in alkaline solution yields indigo. Indoxyl, which is derived from *o*-nitrophenylpropionic acid, appears therefore possibly in the part of an intermediate product in the formation of the colouring matter which it produces when oxidised. Indigo, however, it was argued (Baeyer, I. "Ueber d. Verb. d. Indogruppe," *Ber. Deut. Chem.*

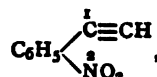
Gesell., xiv., 1741), is of much more complicated nature than indoxyl, giving, for instance, upon reduction substances of the character of complicated hydrocarbons. It is therefore more probable that, in the formation of indigo from indoxyl, a carbon condensation between two molecules of the latter takes place. The constitution of indoxyl, as then regarded by Baeyer, was represented by the following formula:—



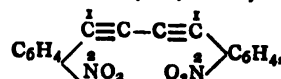
To obtain indigo, by condensation through the medium of oxidation, from indoxyl, the union must take place at the second carbon atom from the benzene ring, since the first carbon binds merely an hydroxyl-group. The hydrocarbon corresponding to such a union, Baeyer concluded, would be—



that is, Glaser's diacetylenylphenyl. To test this view, first the *o*-nitro-compound of the substance must be prepared. Direct nitration does not produce the desired result. *o*-Nitrophenylacetylene was therefore chosen as synthetic material. Numerous ineffectual attempts to unite two molecules of the monoacetylene compound,—



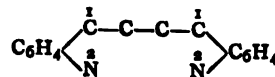
to one molecule of dinitrodiphenyldiacetylene,—



were made. This, notwithstanding, was finally accomplished by oxidation of the copper compound by alkaline solution of potassium ferricyanide. According to the mode of formation, it would be constituted as shown by the formula just given. The double occurrence in this combination of atoms of the group—

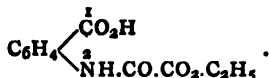


which makes its appearance in the formula of *o*-nitrophenylpropionic acid once, naturally suggested the probability of the formation of di-isatogen by the action of concentrated sulphuric acid in analogy with the formation of isatogen from *o*-nitrophenylpropionic acid under the influence of the same reagent. This actually proved to be the case. In the words of Baeyer:—"This substance claims great interest as being of all artificially prepared substances the one which stands nearest to indigo and is most readily converted into the colouring matter." Simply moistened with ammonium hydrosulphide, it is immediately transformed into indigo, and that quantitatively. The process is further a direct one, the substance instantly becoming blue upon contact with reducing agents without dissolving and without change of form. From the formation of indigo from *o*-nitrophenylacetylene, it appears, therefore, that the production from indoxyl is a double transformation consisting, first, in that the second carbon atoms (from the benzene ring) of two molecules undergo a condensation with the elimination of two atoms of hydrogen; and, in the second place, the body thus formed is converted into indigo by further oxidation. The existence in the indigo molecule of the atomic grouping—

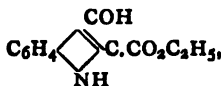


was thus experimentally established, wonderfully confirming Baeyer's deductions based upon the relation of indoxyl to indigo and the complicated structure of the latter. On account of the intrinsic interest of the subject

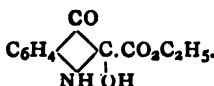
and intimate relation of the compound to indigo, it will be in place to consider further the observations which have led to a knowledge of the constitution of *isatogen* (Baeyer, II. "Ueber d. Verb. d. Indigogruppe," *Ber. Deut. Chem. Gesell.*, xv., 775). It was found that when isatogenic ethylester is reduced in aqueous solution with ferrous salts, that an ester, $C_{11}H_{11}NO_4$, is produced. This same substance is obtained by the gentle and cautious oxidation of indoxyl ethylester. The new substance, indoxanthinic ethylester, is converted by more vigorous oxidisers, such as chromic acid, into ethyloxalylanthranilic acid,—



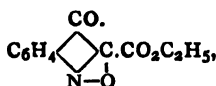
and is, moreover, readily re-converted by reduction into indoxyl ethylester, $C_{11}H_{11}NO_3$, which differ thus from indoxanthinic ethylester by one atom oxygen. The action of nitrous acid upon the latter produces a nitrosamine—an evidence of the presence in the molecule of the imido-group. Assuming for indoxyl ethylester the constitution—



and in view of its close relationship to this compound, the formation of a nitrosamine and the oxidation to ethyloxalylanthranilic acid, the formula of indoxanthinic ethylester would be—



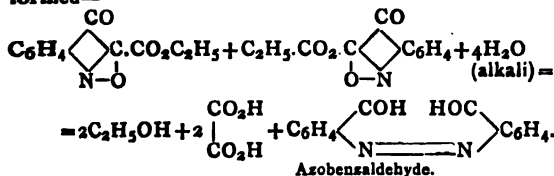
As already stated, the ester is derived by exceedingly cautious reduction of isatogenic ethylester. Representing this body by the formula—



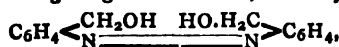
the transition to indoxanthinic ethylester is simply and perfectly explained: the bond between the oxygen and nitrogen atom is dissolved, and, by the addition of two atoms of hydrogen, the oxygen is changed to hydroxyl and the nitrogen to the imido-group as readily seen to be possible by a comparison of the formulae. The characterising supposition is that of the existence of the atomic complex,—



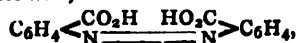
in the isatogen molecule. This is, further, in perfect accord with the fact that by the action of alkali *azo*-benzoic acid (the reactions are not given in detail in the original) is formed—



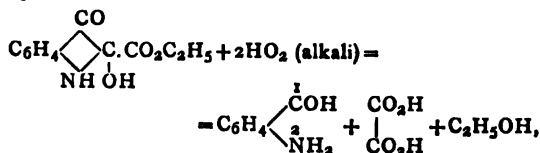
Under the influence of alkali, the aldehyde (two mols.) becomes, according to a general reaction, azobenzylalcohol,—



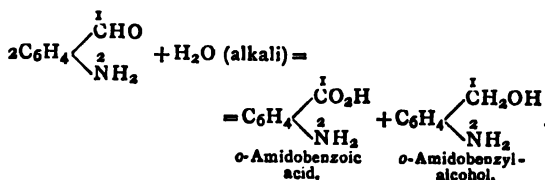
and azobenzoic acid,—



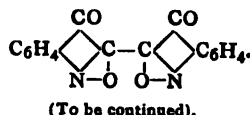
by the addition of two molecules of water. In a similar manner, from indoxanthinic ethylester, *amido*-benzoic acid is produced:—



with the intermediate formation thus of *o*-amidobenzaldehyde. This, as before, yields a corresponding acid and alcohol:—



To *di*-isatogen should therefore be ascribed the formula—



ON TRI-IODIDE OF ARSENIC.

By R. DUPOUY.

TRI-IODIDE of arsenic is a therapeutic substance which was formerly very much used in the treatment of certain cutaneous affections. Of late years this medicament has been administered internally by Dr. R. Saint-Philippe, who has noted the good effects of iodide of arsenic on lymphatic and scrofulous children.

The medicament is supplied as a simple pharmaceutical preparation obtained by treating 1 grm. of iodide of arsenic with 100 grms. of water, and the liquid thus obtained goes under the name of "solution of iodide of arsenic." This name, however, is incorrect, for the water does not behave to the metalloiodide as a simple solvent, but, on the contrary, as a very energetic dissociating agent, and in reality it is not a solution of iodide of arsenic that is used in pharmacy, but rather a solution of arsenious anhydride and hydriodic acid, which are both products of the decomposing action of water on the tri-iodide of arsenic.

While studying the dissociation of iodide of arsenic by water, I have observed that most of the samples supplied commercially leave a residue of variable colour after treatment with water, and further, according to circumstances, the clear liquid obtained was sometimes odourless, but was, on the other hand, sometimes possessed of a very disagreeable smell. In face of such discordant results I thought it as well to make analyses of samples of iodide of arsenic, such as are actually sold, with a view to finding out the causes which make these samples behave in manners so different under the influence of the same chemical reagent, water.

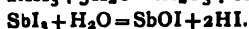
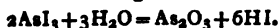
All the samples I was able to obtain belong to one of the four following types:—

Samples, I. The iodide of arsenic appears as a crystalline powder which, examined under the microscope, is seen to consist of hexagonal flakes.

Although this crystalline appearance tells in favour of the purity of the substance, chemical analysis shows that, on the contrary, this sample is not of absolute purity. When, in fact, we triturate 1 grm. of the iodide with 100

grms. of water, there remains a yellow residue which—when separated by filtration and dissolved in hydrochloric acid—gives a precipitate of sulphide of antimony with sulphuretted hydrogen; further, the presence of iodine in this yellow precipitate is easily shown; finally, when working on larger quantities and making a quantitative analysis, we find that the insoluble residue consists of the oxyiodide of antimony, SbOI .

The presence of this impurity is easy to explain when we remember that the arsenic used for the preparation of the tri-iodide sometimes contains antimony: it is evident, therefore, that if we act on this impure arsenic with iodine, there will be formed simultaneously iodide of arsenic and iodide of antimony, which occur, as is well known, under almost identical conditions. To purify the product we must have recourse to solution of the iodide of arsenic in sulphide of carbon; but it must be remarked that not only is iodide of antimony soluble in the same solvent, but, again, that it crystallises like iodide of arsenic in hexagonal flakes. It therefore follows that the crystalline powder contains both iodide of arsenic and iodide of antimony. If we act with water on this mixture we obtain the two following reactions:—



The second equation shows that oxyiodide of antimony is formed, and it is this body that forms the insoluble residue I have already referred to. We see that the water plays the part, to some extent, of a purifying agent, by removing from the iodide of arsenic the antimony it contains, by precipitating this latter body in the form of insoluble oxyiodide.

Finally, it must be remarked that iodide of arsenic, crystallised in sulphide of carbon, and then treated with water, gives a solution having a very disagreeable odour: this odour is that of the sulphide of carbon, which nearly always impregnates bodies crystallised in it.

Samples, II. The samples belonging to this second category occur in the form of masses or plates consisting of fused iodide of arsenic. When treated in the same manner as the last, we obtain a blackish precipitate after treatment with water, a part of which precipitate only is soluble in hydrochloric acid: this soluble part consists of oxyiodide of antimony. The black residue, insoluble in hydrochloric acid, when submitted to ordinary qualitative analysis, is found to be formed in greater part of metalloidal arsenic. These two impurities are a consequence of the method of preparation of melted iodide of arsenic. The oxyiodide of antimony takes its rise from an process analogous to that already described; as for the arsenic, its presence can be explained by the fact that, in reacting with iodine on arsenic, in the dry way, it is very difficult to limit the reaction of the two reacting bodies. One of them, the iodine, being more volatile, partly escapes the action of the arsenic which remains in excess, and which, being insoluble in water, remains as a residue after the treatment of the iodide of arsenic by this liquid.

Samples, III. These samples only differ from the preceding by the nature of the residue after treatment by water. The black insoluble precipitate is almost entirely formed of metalloidal arsenic; there are no traces of oxyiodide of antimony, which indicates that the arsenic which has been used for the preparation of the tri-iodide was free from antimony.

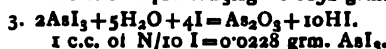
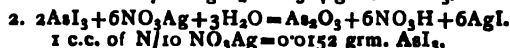
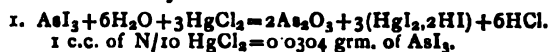
Samples, IV. The appearance of these samples examined is quite different to that of the others; of a deep brown colour, and having a very distinct odour of iodine, they stain the paper on which they may be placed, brown; these characteristics suffice to show the excess of iodine contained in these samples. Further, when treated with water they give a clear solution of a deep brown colour. This colouration sometimes disappears after a short while, for the excess of iodine probably reacts on the products of the dissociation of the iodide of arsenic, giving colourless products.

It results from the above analyses that the iodide of arsenic met with commercially is not chemically pure; it remains to be seen whether the amount of impurity is sufficient to prevent the therapeutic use of commercial iodide of arsenic.

If, for example, we weigh the residues obtained after treatment with water, from the first three groups of samples, we perceive that these precipitates, though very voluminous in appearance, are of very little weight, and the proportion of pure iodide of arsenic is not much diminished by the presence of the above-named impurities.

It is, at any rate, preferable to prepare a pure iodide of arsenic, or else to titrate that met with commercially. To effect this titration, it suffices to remark that iodide of arsenic can be estimated as a soluble iodide, either by Personne's method or by means of the more accurate cyano-argentimetric method described by M. Denigès ("Précis de Chimie Analytique"). Finally, iodide of arsenic can also be titrated as an arsenical compound of minimum oxidation by using iodine solutions, for example.

The three following equations sum up the above three methods, the details of which may be found in text-books of chemical analysis:—



In a future paper I propose to examine the methods of preparing chemically pure iodide of arsenic, and I shall lay stress upon the properties of this body, the chemical history of which is still very incomplete.—*Bull. des Trav. de la Société de Pharm. de Bordeaux*, June, 1900.

CORRESPONDENCE.

STANDARD OF ATOMIC WEIGHTS.

To the Editor of the Chemical News.

SIR,—In accordance with your footnote (*CHEM. NEWS*, lxxxii., p. 66) I enclose my reply to Professor Volhard. I confess I was surprised to find such unanimity in the matter of a standard, for it usually takes a new suggestion many years to receive such general acceptance.—I am, &c.,

JAS. LEWIS HOWE.

Washington and Lee University, U.S.A.,
August 23, 1900.

Herr Prof. J. VOLHARD, Halle-a-S.

DEAR SIR—In reply to your questions in the *CHEMICAL NEWS* of August 10, regarding the standard of atomic weights, &c., I would beg leave to say:—

1. I prefer oxygen = 16 as the standard of atomic weights, for the reasons usually set forth in its favour. Using $\text{O} = 16$ as a standard of atomic weights, and not as a unit, I have not experienced the difficulties sometimes anticipated from a didactic standpoint.

2. I am of the opinion that as many figures should be given in expressing the atomic weights as are established with certainty, and one more which may be uncertain to the extent of several units.

3. I should be glad to have the International Atomic Weight Commission edit the current table of atomic weights on the above basis.

With highest esteem I have the honour to be yours very respectfully,

JAS. LEWIS HOWE.

MISCELLANEOUS.

British Association for the Advancement of Science.—The following are the names of the Officers and Committee of Section B (Chemical Science) at the Bradford Meeting of the British Association:—

President—W. H. Perkin, Jun., LL.D., Ph.D., F.R.S.
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Secretaries—W. M. Gardner; F. S. Kipping, Ph.D., D.Sc.; W. J. Pope; T. K. Rose, D.Sc. (*Recorder*).

Committee—A. H. Allen; Prof. Ossian Aschan; Sir William Roberts-Austen, K.C.B., F.R.S.; L. Blanc; C. H. Bothamley; J. B. Cohen, Ph.D.; T. Fairley, F.R.S.E.; Prof. W. N. Hartley, F.R.S.; A. Lapworth, D.Sc.; D. A. Louis; Prof. Stevenson Macadam, F.R.S.E.; F. H. Neville, F.R.S.; F. W. Richardson; S. Rideal, D.Sc.; Prof. A. Smithells; T. E. Thorpe, F.R.S.

The Papers brought before the Section were as follows:—

President's Address—The Modern System of Teaching Practical Inorganic Chemistry, and its Development.

Report of the Committee on the Teaching of Science in Elementary Schools.

Prof. E. A. Letts, D.Sc. and R. F. Blake—On some Problems connected with Atmospheric Carbonic Anhydride, and on a New and Accurate Method for Determining its Amount.

W. Ackroyd—On the Distribution of Chlorine in West Yorkshire.

W. Ackroyd—On a Limiting Standard of Acidity for Moorland Waters.

T. W. Hime, M.D.—On the Effects of Copper on the Human Body.

Report of the Committee on the Continuation of the Bibliography of Spectroscopy.

Report of the Committee on preparing a New Series of Wave-length Tables of the Spectra of the Elements.

H. B. Dixon, F.R.S., and F. W. Rixon, B.Sc.—On the Specific Heat of Gases at temperatures up to 400°.

Report of the Committee on the Nature of Alloys.

F. H. Neville, F.R.S.—Report on the Chemical Compounds contained in Alloys. To be followed by a discussion.

J. E. Stead—On the Mutual Relations of Iron, Phosphorus, and Carbon when together in Cast Iron and Steel.

J. A. Ewing, F.R.S., and W. Rosenhain, B.A.—On the Crystalline Structure of Metals.

Prof. Barrett, F.R.S.—On the Electric Conductivity of the Alloys of Iron.

C. S. Bradley—On some New Chemical Compounds Discovered by the Use of the Electric Furnace.

Report of the Committee on the Electrolytic Methods of Quantitative Analysis.

H. J. H. Fenton and Miss Gosling—Derivatives of Methylfurfural.

H. J. H. Fenton and H. O. Jones—A simple Method for comparing the Affinities of certain Acids.

W. J. Pope—Recent Developments in Stereochemistry. Followed by a discussion.

A. Lapworth—The Constitution of Camphor. Followed by a discussion.

Julius Bredt—On the Degradation of Camphor.

Prof. Ossian Aschan—On the Camphor Question.

Report of the Committee on Isomeric Naphthalene Derivatives.

Report of the Committee on Isomorphous Derivatives of Benzene.

Report of the Committee on the Relation between the Absorption Spectra and Chemical Constitution of Organic Compounds.

W. R. Hodgkinson—Action of Aluminium Powder on some Phenols and Acids.

L. Limpach and W. R. Hodgkinson—The Direct Preparation of β -Naphthylamine.

C. F. Cross—Interaction of Furfural and Caro's Reagent.

S. Ruhemann and H. E. Stapleton—The Synthesis of Benzo- γ pyrone.

S. Ruhemann and H. E. Stapleton—Combination of Thiophenol and Guaiacol with Esters of Acids of the Acetylene Series.

H. D. Dakin and J. B. Cohen—Chlorination of Aromatic Hydrocarbons.

H. T. Brown, F.R.S.—On some recent work on the Diffusion of Gases and Liquids.

Dr. A. Liebmann—On recent Developments in the Textile Industries. Followed by a discussion.

H. M. Dawson, D.Sc.—On the Influence of Pressure on the Formation of Oceanic Salt Deposits.

Major-General Waterhouse—On the Sensitiveness of Silver to Light.

Dr. J. H. Gladstone, F.R.S., and G. Gladstone—Some Thoughts on Atomic Weights and the Periodic Law.

T. Fairley, F.R.S.E.—On the Relation between the Heating and Lighting Power of Coal Gas.

Dr. J. B. Cohen—On Smoke.

F. W. Richardson—On Bradford Sewage and its Treatment.

W. Leach—On the Treatment of Woolcombers' Effluents.

E. A. Letts, D.Sc., and R. F. Blake—On a New and Accurate Method for Determining Oxygen in Fresh Water, Sea Water, and Sewage Effluents.

W. B. Bottomley, M.A., Ph.D.—On the Utilisation of Sewage Sludge.

Messrs. John J. Griffin and Sons, Limited, have just issued their new Illustrated Catalogue and Price List of Bacteriological Apparatus, of 104 pages. It contains, besides the older and well-known apparatus specially designed for this branch of scientific work, all the latest improvements which have been introduced from time to time. There are also listed a number of pure living cultures and microscopical specimens of various bacteria, which will be found of great use by beginners as a help in the identification of microbes, as well as special sets of apparatus for tubercle and Gram staining, the detection of typhoid, &c. The catalogue concludes with two indices, one for names, the other for numbers.

The Chemical Laboratory Fresenius at Wiesbaden. —During the Summer Term, 1900, there were fifty-two Students on the books. Of these, thirty-three were from Germany, five from Russia, four from England, two from Austria and two from France, one from Luxemburg, one from Sweden, one from Italy, one from Spain, one from the United States of America, and one from Rhodesia in South Africa. There were three Assistants in the Instruction-Laboratory, and twenty-four in the Versuchsstationen (private laboratories). In the certified body of Teachers there has been no change. To this body belong the Directors, Prof. Dr. H. Fresenius, Prof. Dr. W. Fresenius, Prof. Dr. E. Hintz, and also Dr. Med. G. Frank, Dr. W. Lenz, Dr. L. Grünhut, and T. Huber (Architekt). The next Winter Term begins on October 15th. During the Summer Term, 1900, besides the scientific researches, a great number of analyses were undertaken in the different departments of the Laboratory (Versuchsstationen) on behalf of trade, manufacture, mining, agriculture, and hygiene.

The Decomposition of Silicates by Boric Anhydride.—P. Jannasch and H. Weber.—In the year 1895 Jannasch first proposed this method, which has since been shown to be insufficient for the attack of certain difficultly decomposable silicates, such as diathene and topaz. The authors have observed, however, that by sufficiently raising the temperature this process is capable of a general application. It is rapid, and allows of the estimation of all the elements of the mineral, including the alkalis, on a single sample. The finely powdered material is

mixed with 15 or 20 parts of powdered Bo_2O_3 . The whole is heated in a platinum crucible, first over a Bunsen burner, then over a blowpipe flame until in tranquil fusion. After partial cooling, 5 or 6 parts more of Bo_2O_3 are added, and the whole calcined over an oxyhydrogen blowpipe until the mass is completely transparent; this requires about a quarter of an hour. The gas from five to six ordinary taps must be brought to the blowpipe, and the oxygen injected by means of the second tube until the flame is no longer luminous. The contents of the crucible, which should remain limpid on cooling, are taken up immediately with a mixture of HCl and methylic alcohol, and heated gently on a water-bath until dry. This evaporation is repeated a second time with the same mixture; the expulsion of the Bo_2O_3 is very rapid. The analysis is then continued in the ordinary manner. It is as well to note that, in the case of fluoriferous silicates, the whole of the fluorine is given off in the state of BoF_3 , without any loss of silica, if we take care to use a sufficient excess of Bo_2O_3 . The method appears to be equally suitable in the case of fluosilicates and natural fluorides. — *Berichte*, vol. xxii., p. 1670.

The Ethylenediaminic Compounds of Palladium.—N. S. Koursanof and N. J. Gvosdaref.—The researches of Joergensen having shown that the compounds of ethylenediamine with the salts of platinum possess a high degree of stability, it appeared to be of interest to examine the analogous compounds of palladium, which, by the properties of its complex salts, forms the connecting link between the platinum metals and the Cu, Ni, Ag group. By the action of ethylenediamine on chloropalladite of potassium, K_2PdCl_4 , a rose-coloured precipitate is first formed, the composition of which is probably $\text{PdCl}_4 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2$, and which re-dissolves in an excess of $\text{C}_2\text{H}_4(\text{NH}_2)_2$, giving a yellowish solution. On evaporation, this latter leaves colourless prismatic crystals of the composition $\text{PdCl}_4 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2$. Contrary to what takes place with the corresponding platinum compound, this body is decomposed by dilute HCl; it loses 1 molecule of $\text{C}_2\text{H}_4(\text{NH}_2)_2$, and is transformed into $\text{PdCl}_4 \cdot \text{C}_2\text{H}_4(\text{NH}_2)_2$ in the form of small yellowish needles slightly soluble in water (1 part in 1000 parts of water at 17°). If we digest this new compound with an aqueous solution of thio-urea, $\text{C}_2\text{H}_4(\text{NH}_2)_2$ is again displaced, and we obtain red crystals of $\text{PdCl}_4 \cdot 4\text{CSN}_2\text{H}_4$, but we have not observed the formation of the complex body containing both $\text{C}_2\text{H}_4(\text{NH}_2)_2$ and CSN_2H_4 . Concentrated HCl easily dissolves $\text{PdCl}_4 \cdot \text{C}_2\text{H}_4(\text{NH}_2)_2$, giving a yellowish-brown liquid, which, on evaporation or cooling, deposits yellowish-brown plates, appearing violet by reflected light, which appear to result from the union of 2HCl with the preceding salt, and have the composition of the chloropalladite of ethylenediammonium, $\text{PdCl}_4(\text{C}_2\text{H}_4\text{N}_2\text{H}_6)$, (type $\text{PdCl}_4\text{R}'$). This chloropalladite is soluble in water; the solution is of a yellowish-brown colour, but it gradually decomposes and deposits $\text{PdCl}_4 \cdot \text{C}_2\text{H}_4(\text{NH}_2)_2$. Heat accelerates this decomposition, the presence of HCl prevents it. The chloropalladite can be re-crystallised in concentrated HCl; we thus have an interesting case of mobile equilibrium dependent on the temperature and the concentration of the HCl: $\text{PdCl}_4 \cdot \text{C}_2\text{H}_4(\text{NH}_2)_2 + 2\text{HCl} \rightleftharpoons \text{PdCl}_4(\text{C}_2\text{H}_4\text{N}_2\text{H}_6) + 2\text{HCl}$. This decomposition is analogous to the reaction observed by Anderson with chloroplatinate of pyridine.—*Fourm. Soc. Phys. Chim. R.*, vol. xxxi., p. 691.

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NOTICE.

The STUDENTS' NUMBER of the CHEMICAL NEWS will be published on Friday, September 21st. Gentlemen holding official positions in the Universities, Medical Schools, &c., of the United Kingdom, where Chemistry and Physical Science form a part of the education, who have not yet forwarded the necessary information to our Office for publication in that Number, will confer a favour by sending it with the least possible delay.

Advertisements for this Number should reach the Office not later than Wednesday, Sept. 19th.

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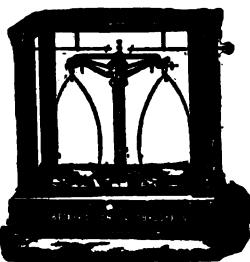
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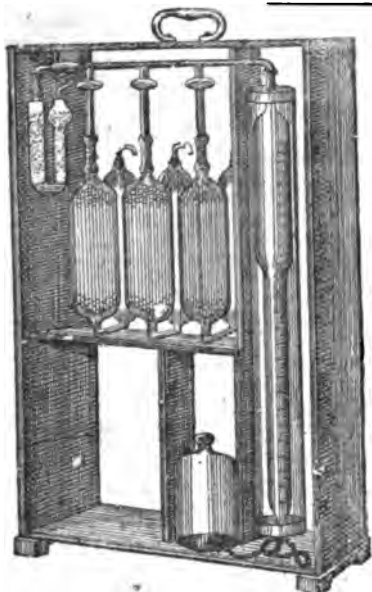
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2130.

(STUDENTS' NUMBER).

ADDRESS TO STUDENTS.

WITH the advent of October the various Schools and Colleges for Scientific and Medical instruction again open their doors to students.

Every facility and encouragement is offered to those who care to take advantage of their opportunities. Many students are returning to familiar scenes and faces after a long vacation, in which they have been recuperating their energies and fitting themselves for further efforts in their advance towards the "degree" which will be, at any rate, the outward and visible result of their labours, if successful. Others, fresh from school, will find a great change in more ways than one. College life is much more free than the stricter routine of our school days; the Student is left more to the promptings of his own inclination, and is able to shirk many duties without apparently being any the worse off for it. It is to these younger students, commencing their careers, practically with the dawn of the twentieth century, that we would specially point out the urgent necessity of neglecting no opportunities; these, if not taken at the flood, may never recur again, and the words "too late" will be but a sad solace to a wasted youth.

As Sir Wm. Turner pointed out in his recent address to the British Association at Bradford, "diligence and accuracy" are fundamental qualities in scientific research; "by their application new facts are discovered and tabulated, but to decide on their true significance a well-balanced mind and the exercise of prolonged thought and reflection are needed." These latter qualifications will accrue later on in life, but "diligence and accuracy" should be the watchword of the student in these his college days.

UNIVERSITIES AND COLLEGES.

UNIVERSITY OF LONDON.

CANDIDATES for any Degree in this University must have passed the Matriculation Examination. No exemption from this rule is allowed on account of Degrees obtained or Examinations passed at any other University. This and all other Examinations of the University, together with the Prizes, Exhibitions, Scholarships, and Medals depending upon them, are open to Women upon exactly the same conditions as to Men.

There are two Examinations for Matriculation in each year; one commencing on the second Monday in January, and the other on the second Monday in June.

The Examination is conducted by means of Printed Papers; but the Examiners are not precluded from putting, for the purpose of ascertaining the competence of the candidates to pass, *viva voce* questions to any candidate in the subjects in which they are appointed to examine. These Examinations may be held not only at the University of London, but also, under special arrangement, in other parts of the United Kingdom, or in the Colonies.

Every candidate for the Matriculation Examination must, not less than five weeks before the commencement of the Examination, apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the commencement of the Examination, accompanied by a Certificate showing that the candidate has completed his sixteenth year, and by his Fee for the Examination. As no candidate can be admitted after the List is closed, any candidate who may not have received a Form of Entry within a week after applying for it must communicate immediately with the Registrar, stating the exact date of his application and the place where it was posted.

Every candidate entering for the Matriculation Examination for the first time must pay a Fee of £2 to the Registrar. If a candidate withdraws his name, or fails to present himself at the Examination, or fails to pass it, the Fee shall not be returned to him, but he shall be allowed to enter for any subsequent Matriculation Examination upon payment, at every such entry, of an additional Fee of £1, provided that he comply with the Regulations in the preceding paragraph.

Candidates are not approved by the Examiners unless they have shown a competent knowledge in each of the following subjects:—Latin (two papers); English (two papers). Any one of the following Languages or Sciences:—Greek, French, German, Sanskrit, or Arabic; Mathematics (two papers); General Elementary Science (two papers); Elementary Mechanics, Chemistry, Sound, Heat and Light, Magnetism and Electricity, Botany.

A Pass Certificate, signed by the Registrar, will be delivered to each successful candidate after the Report of the Examiners has been approved by the Senate.

The first six candidates in the Honours Division of not more than twenty years of age at the commencement of the Examination will respectively receive an Exhibition or a Prize as follows:—The first of such candidates will receive an Exhibition of £30 per annum for the next two years. The second will receive an Exhibition of £20 per annum for the next two years. The third will receive an Exhibition of £15 per annum for the next two years; such exhibitions are payable in quarterly instalments provided that on receiving each instalment the Exhibitioner declares his intention of presenting himself either at the two Examinations for B.A., or at the two Examinations for B.Sc., or at the Intermediate Examination in Laws, or at the Preliminary Scientific and Intermediate Examinations in Medicine, within three academical years from the time of his passing the Matriculation Examination. The fourth will receive a prize to the value of £10 in books, philosophical instruments, or money; and the fifth and sixth will each receive a prize to the value of £5 in books, philosophical instruments, or money.

Any candidate who may obtain a place in the Honours Division at the Matriculation Examination in January is admissible to the Intermediate Examination either in Arts or in Science in the following July.

INTERMEDIATE EXAMINATION IN SCIENCE.

The Intermediate Examination in Science will commence on the second Monday in July.

No candidate (with the exception of such as have obtained Honours at the Matriculation Examination in the preceding January) is admitted to this Examination within one academical year of the time of his passing the Matriculation Examination.

The Fee for this Examination is £5.

Examination for Honours.

Candidates for Honours in Chemistry will be examined in Inorganic Chemistry, treated more fully than in the Pass Examination. This Examination will consist of two printed papers and a practical examination.

In the Examination for Honours, the Candidate, not being more than 22 years of age at the commencement of the Pass Examination, who most distinguishes himself will receive an Exhibition of £40 per annum for the next two years.

B.Sc. EXAMINATION.

The B.Sc. Examination will be held on the fourth Monday in October.

Candidates for this Examination are required to have passed the Intermediate Examination in Science at least one academical year previously.

The Fee for this Examination is £5.

Examination for Honours.

For the examination for Honours in Chemistry two papers will be set and a two days' practical examination.

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DOCTOR OF SCIENCE.

The examination for the Degree of Doctor of Science takes place annually within the first twenty-one days of June.

No candidate is admitted to the examination for the Degree of D.Sc. until after the expiration of two Academical Years from the time of his obtaining the Degree of B.Sc. in this University.

Every candidate for this Degree must state in writing the special subject within the purview of the Faculty of Science, as set out in the Programme of the B.Sc. Examination, upon a knowledge of which he rests his qualification for the Doctorate; and with the Form of Entry he shall transmit an original Dissertation or Thesis (at least six copies), printed, type-written, or published in his own name, treating scientifically some special portion of the subject so stated, embodying the result of independent research, or showing evidence of his own work, whether conducted independently or under advice, and whether based on the discovery of new facts observed by himself, or of new relations of facts observed by others, or, generally, tending to the advancement of Science. Every candidate may further specify any printed contribution or contributions to the advancement of Science which he has at any time previously published. If the Dissertation or Thesis be approved by the Examiners, the candidate shall be required to present himself at the University upon such day or days within the first twenty-one days of June as may be notified to him, and shall, at the discretion of the Examiners, be further tested, either orally or practically, or by printed questions or by all of these methods, with reference both to the special subject selected by him and to the Thesis.

PRELIMINARY SCIENTIFIC (M.B.) EXAMINATION.

This Examination takes place twice in each year, once, for Pass and Honours, commencing on the second Monday in July; and once for Pass Candidates only, commencing on the third Monday in January.

No candidate shall be admitted to this Examination unless he shall have passed the Matriculation Examination. Not less than five weeks before the commencement of the Examination he must apply to the Registrar for a Form of Entry, which must be returned not less than four weeks before the Examination, accompanied with the candidate's fee.

The Fee for this examination is Five Pounds.

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Every Student must reside in one or other of the Colleges or Halls, or in licensed lodgings, for a period of three years. Students of Chemistry can obtain the degree of B.A. by passing preliminary examinations in Arts and in Science, and a final Honour examination in Chemistry. Chemistry may also be taken as part of the examination for a Pass degree. Graduates of other Universities suitably qualified can obtain the degree of Bachelor of Science after an approved course of study or research and two years' residence.

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More detailed information may be obtained from the Examination Statutes; the Student's Handbook to the University; and from the professors and college tutors.

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Jacksonian Professor of Natural and Experimental Philosophy—J. Dewar, M.A., F.R.S.

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A graduate of another University may be admitted as an "advanced" student, and obtain a degree after two years' residence in the University.

The scholarships, ranging in value from £20 to £100 a year, are chiefly given for mathematical and classical proficiency. Scholarships, or Exhibitions, are given for Natural Science in King's, Trinity, St. John's, St. Peter's, Clare, Trinity Hall, Queen's, Jesus, Christ's, Emmanuel, Sidney, Pembroke, Caius, and Downing Colleges; the dates of the examinations vary, but are always fully advertised.

The Chemical Laboratory of the University is open daily for the use of the Students. The Demonstrators attend daily to give instructions. A list of the lectures is published annually, in June, in a special number of the *Cambridge University Reporter*, which may be had from the Cambridge Warehouse, in Paternoster Row, or through any bookseller.

Non-collegiate Students are allowed to attend certain of the College Lectures and all the Professors' Lectures, and have the same University status and privileges as the other Students. Full particulars may be obtained by forwarding a stamped directed envelope to the Assistant Registrar, Cambridge, or from the *Cambridge University Calendar*.

UNIVERSITY OF DUBLIN.

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3. *Metallurgy.*—A Course for Engineering and Technical Students.

The Laboratories are open every day from 10 to 5 o'clock (except Saturdays, when they close at 1 o'clock).

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By recent decrees, Prizes in Chemistry and Physics will be given in future at the October (Arts) Entrance Examination, and also at the Terminal Examinations of the Junior and Senior Freshmen Years.

Similarly, two Science Scholarships will be obtainable by Freshmen, and tenable for five years. These Foundation Scholarships are paid £20 per annum, have free commons, and their chambers and tutorial fees are at half the usual rates.

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Demonstrator of Practical Chemistry—Herbert Jackson, F.C.S.

Assistant Demonstrator—P. H. Kirkaldy, F.C.S.

The Academical Year consists of Three terms. The days fixed for the Admission of New Students in the Academical Year 1900-1901 are October 4, January 17, and April 25.

Students of the First Year are admitted to the Course of Theoretical and Applied Chemistry. The Course commences with a view of the conditions suitable for the production of Chemical Phenomena, after which the laws of Chemical Attraction are discussed, and the Non-metallic Elements and their principal compounds are described. The Metals and their principal compounds are next examined, care being taken to point out the applications of the Science to the Arts; and the processes of the different Manufactures and of Domestic Economy are explained and illustrated. Examinations of the Class, both *visd voce* and by written papers, are held at intervals during the course at the usual Lecture hour.

Second Year.—Students attend in the Laboratory twice a week, and they go through a course of Manipulation in the most important operations of Chemistry, including the first steps of Analysis. Any Student of this Division may be admitted to this Class at any period of his study on payment of an extra fee.

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Lecturer—Prof. J. M. Thomson, F.R.S., F.C.S.

In addition to the regular College Course in Photography occasional classes may be formed. For further particulars application should be made to Prof. Thomson.

EVENING CLASSES.

Classes for Evening Instruction in various subjects are held during the Winter Session.

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Professor—William Ramsay, LL.D., F.R.S., &c.

Assistants—Morris Travers, D.Sc., Alexander Kellas, B.Sc., and E. C. C. Baly, F.I.C.

The Session is divided into three Terms, as follows:—First Term, from Tuesday, October 2nd, till Friday, December 21st; Second Term, from Tuesday, January 15th, till Friday, March 29th; Third Term, from Tuesday, April 23rd, till Friday, June 28th. Class Examinations begin on June 17th.

Junior Courses of Inorganic Chemistry.

First Term: Tuesday, Thursday, and Saturday at 10., Latter half of Second and Third Term: Tuesday, Thursday, and Saturday. Fee:—£4 4s.

These Courses will each consist of about thirty lessons, partly theoretical and partly practical, on the non-metallic elements. Frequent exercises will be given.

Senior Course of Inorganic Chemistry.

First and Second Terms: The Class meets four times a week, on Mondays, Wednesdays, Fridays, and Saturdays, at 9, for Lectures, Examinations, and Exercises.

Fees:—For the Course, £7 7s.; Perpetual, £9 9s.; for the First or Second Term, £4 4s.

This Course and the Practical Class cover the subject as prescribed for the Preliminary Scientific (M.B.) and Int. Examination in Science of the University of London.

For the Preliminary Scientific Examination Students who take the three subjects for that examination in July attend during the First and Second Terms.

Advanced Course of Chemistry.

Second and Third Terms.—The class meets twice a week, on Tuesdays and Thursdays, at 9, beginning on January 15. The hour will be altered by special arrangement with the class if necessary.

Fee:—For the Course, £3 3s.; for a Term, £2 2s.

This Course will be found suitable for those about to proceed to graduation as Bachelor of Science in London University, and to those who intend to choose Chemistry as a profession. Such students should also work in the Laboratory during as many hours as they can spare.

Organic Chemistry.

Mondays and Wednesdays, at 9, during the session.

This Course of Organic Chemistry is intended for those who are studying the subject from a scientific standpoint. Candidates for Honours at the Int.M.B. are, however, recommended to attend this Course instead of the Special Summer Course.

The Course includes the subjects required at the B.Sc. Examination, Pass and Honours; but no previous acquaintance with Organic Chemistry will be expected of those joining the Class.

Fee:—For the Course, £6 6s.; for a Term, £2 12s. 6d.; for a Second Course, £3 3s.

Practical Classes.

Practical Classes in Inorganic and Organic Chemistry are conducted by the Assistants.

Analytical and Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., Saturdays excepted, from October until the middle of July, with a short recess at Christmas and at Easter.

Fees: for the Session, £26 5s.; six months, £18 18s.; three months, £10 10s.; one month, £4 4s.

Three specified days a week:—for the Session, £15 15s.; six months, £11 11s.; three months, £6 6s.; one month, £2 12s. 6d., exclusive of expense of materials. Students may enter at any period of the Session.

The Laboratory Course includes the Practical Chemistry required at the following Examinations of the University of London:—Prel. Sci. (M.B.), Intermediate M.B., Intermediate Science, B.Sc.

When accompanied by, or preceded by, attendance on the Lectures on Inorganic and Organic Chemistry, the Laboratory Course qualifies Students in the application of Chemistry to Manufactures, Metallurgy, Medicine, or Agriculture, &c.

There is also a Chemical Library containing the chief Journals and Standard Works on Chemistry.

Certificates of Honour are granted to competent Students on the work done during the Session. The Tuffnell Scholarship (£100 for two years) will also be competed for in the Session 1900-1901; also the Cloth-worker's Scholarship of £30.

ROYAL COLLEGE OF SCIENCE AND ROYAL SCHOOL OF MINES.

Professor—W. A. Tilden, D.Sc., F.R.S.

Assistant Professor—W. P. Wynne, D.Sc., F.R.S.

Demonstrators—H. Chapman Jones and M. O. Forster, Ph.D., B.Sc.

Assistants—G. S. Newth, G. T. Morgan, B.Sc., and J. A. Craw.

The Royal College of Science at South Kensington is intended, primarily, for the instruction of teachers, and of students of the industrial classes selected by competition in the examinations of the Science and Art Department. The Royal School of Mines is incorporated with the Royal College of Science. Students entering for the Associateship of the Royal School of Mines obtain their general scientific training in the Royal College of Science. The instruction in the Royal College of Science is arranged in such a manner as to give the Students a thorough training in the general principles of Science, followed by advanced instruction in one or more special branches of Science. The Associateship is granted in certain divisions or lines of study. Students who go through any one of the prescribed courses of instruction and pass the necessary Examinations receive a Certificate of Associateship of the Royal College of Science, or of the Royal School of Mines. Students who are not candidates for the Associateship are permitted to enter as occasional students in one or more special branches of science, and on passing the examination receive a Certificate to that effect. The Associateship of the Royal College of Science is given in one or more of the following divisions:—Mechanics, Physics, Chemistry, Biology, and Geology, and the Associateship of the Royal School of Mines in Metallurgy and Mining.

The course of instruction, which lasts for three years, is the same for all the divisions during the first year, after which it is specialised in accordance with the Scheme detailed in the Prospectus of the School.

Examinations are held at the end of each course of instruction and at such other periods as may be found necessary. On the results of these examinations the successful candidates are arranged in two classes, first and second. There are also "Honours" examinations for the subjects of the third year, the successful candidates being placed in order of merit. A student obtains the Associateship who passes in all the subjects of the first year, and, in the

second and third year, those subjects prescribed as necessary for the division in which he seeks to obtain his Associateship. A student who goes through the prescribed course of instruction in any subject and passes the necessary examinations receives a certificate to that effect.

Students who do not wish to attend the lectures are admitted for short periods to the laboratories, at the discretion of the Professors. The fees for the laboratories are £4 per month.

Students not entering for the Associateship are admitted to any particular course of study, so far as there is room, on payment of the fees shown in the following table:—

	Lectures.	Laboratory.
Chemistry	£ 3	£ 13
Physics	5	12
Biology with Botany	5	12
Geology with Mineralogy	4	8
Mechanics	4	6
Metallurgy	2	13
Mining	4	
Astronomical Physics	2	3

Mathematics and Mechanical Drawing, £3 per term. Model and Freehand Drawing, £1 per term. Descriptive Geometry, £3 per session. Mine Surveying, £10.

The fees for the first two years amount to about £75, and for the remainder of the course for the Associateship they vary from £30 to £40.

Several valuable Exhibitions, Scholarships, and Prizes are attached to the studentship.

Summer Courses for Teachers.—Short courses of instruction are given annually, about July, in different branches of science for the benefit of teachers of science schools in the country. The courses last three weeks. About 250 teachers are admitted to them, and they receive third class railway fare to and from South Kensington, and a sum not exceeding £3 towards their expenses. (See Science and Art Directory).

Working Men's Lectures.—Notification of these will be given in the newspapers.

THE SCHOOL OF THE PHARMACEUTICAL SOCIETY OF GREAT BRITAIN.

The Fifty-ninth Session will commence on Monday, October 1st, 1900.

Professors—Chemistry, J. Norman Collie, Ph.D., F.R.S. (Dean); Botany, J. Reynolds Green, Sc.D., F.R.S., F.L.S.; Pharmaceutics, Henry G. Greenish, F.I.C., F.L.S.

A Course of Lectures on Physical, Inorganic, and Elementary Organic Chemistry commences in October and terminates at the end of June. An Advanced Course of Lectures begins in October and extends to the end of March. These Lectures are adapted to the requirements of Pharmaceutical and Medical Students, and also those who are proceeding to degrees at the University of London, or who are preparing for the examinations of the Institute of Chemistry.

Entries may be made for single classes. Certificates of attendance at the two Courses of Lectures on Chemistry and at the Chemical Laboratories are accepted as evidence of chemical training by the Institute of Chemistry in connection with the Examinations for the Associateship, and also by the conjoint Board of the Royal Colleges of Physicians and Surgeons, as well as by other examining bodies.

Prospectuses and further information may be obtained from Mr. Richard Brembridge, Secretary and Registrar, 17, Bloomsbury Square, London, W.C.

UNIVERSITY COLLEGE OF WALES, ABERYSTWYTH.

UNIVERSITY OF WALES.

Professor—H. Ll. Snape, D.Sc. (Lond.), Ph.D. (Göttingen), F.I.C.

Demonstrators—H. Hibbert, M.Sc. (Vic.), and A. Brooke, Ph.D. (Strassburg).

Lecturer in Agricultural Chemistry.—J. Alan Murray, B.Sc. (Edin.).

The College is open to male and female students above the age of sixteen years. The Session commences on Tuesday, October 2, on which day all Students will be expected to meet the Professors in the Examination Hall of the College.

Lecture Courses.—(1) Matriculation Course; three lectures weekly during the Michaelmas and two weekly during the Lent and Easter Terms. (2) Intermediate Science Course; four lectures weekly during the Lent and Easter Terms. (3 and 4) B.Sc. Courses; A, three lectures weekly on Organic Chemistry; B, two lectures weekly on Chemical Theory. (Courses A and B will generally be given in alternate Sessions; for 1900-1901, Course B). (5 and 6) Courses in Agricultural Chemistry. For students in their first year, 3 lectures, and for those in their 2nd year, 2 lectures weekly during the Michaelmas and Lent terms.

Laboratory Courses.—The Laboratory is open daily from 10 a.m. to 1 p.m., and from 2.15 to 5 p.m., except on Wednesdays and Saturdays. Classes for the Systematic Study of Qualitative and Quantitative Analysis will be formed, and Special Courses will be arranged for those who intend to follow Medicine or Pharmacy, or any one particular branch of Applied Chemistry, always provided that such Students possess the requisite knowledge of Theoretical Chemistry. The hours will be arranged, as far as possible, to suit the requirements of the individual Student.

The College is recognised by the University of Edinburgh and the Royal University of Ireland, and by the Colleges of Physicians and Surgeons of England, Scotland, and Ireland as an institution at which the instruction necessary for their respective Diplomas in Medicine, in Chemistry, Physics, and Biology may be given. One year for graduation in Medicine and two years for graduation in Science may be spent at Aberystwyth.

Fees.—The Fee for the whole Session, if paid in advance, is £10; if paid by Single Terms, for the first term of attendance in each Session, £4; for the second term, £3 10s.; for the third term, £3. These composition fees enable the Student to attend any or all the Classes of the College, with the exception that a small extra fee is charged for Laboratory Instruction. Thus, for Practical Chemistry, the additional fee is, for six hours' work per week, 12s. 6d. per term, and for twelve hours, 25s. per term. The fees for those who desire to spend several days weekly in the laboratory may be learned on application to the Registrar. Fee for a single Lecture Course £1 per term.

Scholarships and Exhibitions varying in value from £10 to £40 per annum will be offered for competition at examinations which commence on September 18, and exhibitions are awarded at the end of the Session on the results of the class examinations.

Intending Students requiring further information are recommended to write to the Registrar for a copy either of the General Prospectus or of one of the Special Prospectuses issued for the Agricultural and Normal Departments.

UNIVERSITY COLLEGE OF NORTH WALES, BANGOR.

A CONSTITUENT COLLEGE OF THE UNIVERSITY OF WALES.

Chemistry.—Professor, James J. Dobbie, M.A., D.Sc. Demonstrator, Alexander Lauder, B.Sc., F.C.S. Assistant Lecturer in Agricultural Chemistry, F. V. Dutton. Scholar Assistant, Alice E. Smith.

Physics.—Professor, E. Taylor Jones, D.Sc.

The Session opens October 2nd, 1900. All regular classes are open to men and women students above the age of 16 years. The following Courses of Lectures will be given.

Matriculation Course.—Subjects: Those prescribed for

the Matriculation Examination of the University of Wales. Fee for the Session £3 3s.

Intermediate Course.—Inorganic Chemistry and Elementary Physical Chemistry. Fee for the Session, £3 13s. 6d.

B.Sc. Course.—Organic Chemistry. Fee for the Session, £3 3s.

Agricultural Chemistry.—Fee, £2 2s.

Laboratory Courses.—The laboratory is open on five days of the week from 10 a.m. to 4 p.m. for instruction in Chemical Analysis and in the Application of Chemistry to Medicine and the Industrial Arts. Fees: six hours per week, £1 1s. per Term; twelve hours, £2 2s.; eighteen hours, £3 3s.; twenty-four hours, £4 4s. Composition Fee for all Laboratory Classes of the Intermediate Science Course taken in one year, £4 4s.

The Chemistry, Botany, Zoology, and Physics Courses are recognised for Medical graduation in the Universities of Edinburgh and Glasgow, and students can make one *Annus medicus* at the college. The Science Courses are recognised for part of the science degree course of the University of Edinburgh.

UNIVERSITY COLLEGE OF SOUTH WALES AND MONMOUTHSHIRE, CARDIFF.

Professor.—C. M. Thompson, M.A., D.Sc., F.C.S.

Demonstrators.—E. P. Perman, D.Sc., F.C.S., and A. A. Read, F.I.C., F.C.S.

The Session commences October 8th, and terminates on June 28th, and is divided into three terms.

The Junior Course (delivered during the Michaelmas term only) consists of about 50 lectures, and will cover the subjects prescribed for the Matriculation examinations of the University of Wales and the University of London. Fee, £2 2s. A revision class is held in the Summer term.

The Intermediate Course consists of about 80 lectures held during the Lent and Summer terms in continuation of the Junior Course, and is the qualifying course for the Intermediate Examination of the University of Wales. Together with laboratory practice, it will cover the subjects required for the Intermediate Examination in Science and the Prel. Sci. (M.B.) Examination of the University of London. Fee, £4 4s.

The Senior Course consists of some 90 lectures on Organic Chemistry; Fee, £3 3s.

A course of 20 lectures on Qualitative Analysis and a short course on Organic Chemistry will also be given.

The following lectures on Metallurgy will be given by Mr. Read:—10 lectures on Fuel; Fee, 10s. 6d. 20 lectures on General Metallurgy; Fee, £1 1s. 30 lectures on the Manufacture of Iron and Steel; Fee, £1 1s. A practical course on Iron and Steel Analysis will also be held, and practical instruction in Dry Assaying will be given in the Metallurgical Laboratory, which is fitted with the necessary furnaces and apparatus.

In the laboratory each student works independently, so that the course of study may be adapted to the requirements of the individual. Hours, 9 to 1 and 2 to 5; Saturday, 9 to 1. Fees—Six hours per week, £3 3s. per session; twelve hours, £2 2s. per term; eighteen hours, £3 3s. per term; twenty-four hours £4 4s. per term.

Registered medical students can prepare for the Intermediate M.B. Examination of the University of London, and spend three out of their five years of medical study in Cardiff. Medical students wishing to graduate at a Scottish University, or preparing for a Conjoint Board Surgical and Medical Diploma, or for the Diploma of the Society of Apothecaries, can spend two years in Cardiff. For further information see the prospectus of the Faculty of Medicine, which may be obtained from the Registrar.

The College is recognised as an institution at which two years of the course for the degree of Bachelor of Science of the University of Edinburgh may be spent.

Students by making a payment of £10 at the commencement of each session may compound for all lecture fees for the whole session. Laboratory fees are not in-

cluded in the composition fee, but Students preparing for the Science Examinations of the University of Wales and of the University of London may, by making a payment of £13 13s. at the commencement of each Session, compound for both Lecture and Laboratory Fees during the Session.

At the entrance examination in September, and the annual examination in June, several scholarships and exhibitions are awarded. Great importance is attached to special excellence in one subject.

The College Prospectus, and also further information as to scholarships, may be obtained from the Registrar.

A Hall of Residence for Women Students is attached to the College.

UNIVERSITY COLLEGE, BRISTOL.

Professor of Chemistry—Sydney Young, D.Sc., F.R.S.

Lecturer—Francis E. Francis, B.Sc., Ph.D.

Demonstrator—E. B. Ludlam, B.Sc.

The session 1900-1901 will begin on October 2. Lectures and classes are held every day and evening throughout the Session. In the Chemical Department lectures and classes are given in all branches of theoretical chemistry, and instruction in practical chemistry is given daily in the chemical laboratory. The department of experimental physics includes various courses of lectures arranged progressively, and practical instruction is given in the physical and electrical laboratories. The Department of Engineering and the Constructive Professions is designed to afford a thorough scientific education to students intending to become engineers, or to enter any of the allied professions, and to supplement the ordinary professional training by systematic technical teaching. This department includes courses specially arranged for students intending to become civil, mechanical, electrical, or mining engineers, surveyors, or architects. Those who attend the mechanical engineering course enter engineering works during the six summer months, and, in accordance with this scheme, various manufacturing engineers in the neighbourhood have consented to receive students of the College into their offices and workshops as articulated pupils at reduced terms. Medical education is provided by the Faculty of Medicine of the College. Several Scholarships are tenable at the College. Full information may be obtained from the Secretary.

DAY LECTURES.

Inorganic Chemistry.

The Courses treat of the principles of Chemistry, and of the Chemistry of the Non-Metals and Metals.

Junior Course.—Two Lectures a week will be given during the Session. Fee, £4 4s.

General Elementary Science (London Matriculation).—A course of about 16 Lectures will be delivered in the Third Term. Fee, including Physics (1st and 2nd Terms), £5 5s.

Special Course.—A special course of Lectures is also given to Engineering Students.

Intermediate Course.—Three Lectures a week will be given throughout the Session. Fee, £5 5s. There will be tutorial classes in connection with the Junior and Senior Courses.

Advanced Course (Parts I. and II.).—One Lecture a week in each part will be given throughout the Session. Fee for each course, £2 12s. 6d.

Organic Chemistry.

This Course will relate to the more important groups of the Compounds of Carbon.

Two Lectures a week will be given throughout the Session. Fee, £3 3s. An advanced course of lectures will also be given one day a week during the session. Fee, £2 12s. 6d.

Practical Chemistry.—Laboratory Instruction.

The Laboratory will be open daily from 10 a.m. to 5 p.m., except on Saturdays, when it will be closed. Instruction will be given in the Laboratory in all branches of Practical Chemistry, including Qualitative and Quantitative Inorganic and Organic Analysis, the preparation of

Chemical Products, Metallurgy, and Inorganic and Organic Research. Special facilities will be afforded to those who desire to study Practical Chemistry as applied to the different processes employed in the Arts and Manufactures. Fees in Guineas—

	5 Days a Week.	4 Days a Week.	3 Days a Week.	2 Days a Week.	1 Day a Week.
Per Session ..	15	12½	10	7½	5
„ Two Terms ..	11	9	7½	5½	3½
„ One Term ..	7	6	4½	3½	2½

Students may arrange to divide their days of laboratory work into half-days.

Chemical Scholarship.—Among others, a Chemical Scholarship of £25 is offered for competition.

EVENING LECTURES.

Two courses of Lectures will be delivered during the First and Second Terms; they will be devoted to the consideration of the general Principles of Chemistry and Chemical Physics and the Chemistry of Non-Metallic and Metallic Elements. Special attention will be paid throughout to those products which have a practical application in the Arts and Manufactures. Fee for each course, 7s. 6d.

General Elementary Science.—A course of Lectures, primarily intended for Candidates for Matriculation, will be given during the First Term. Fee (including Physics), 7s. 6d.

Practical Chemistry.—Laboratory Instruction.—The Laboratory will be open two evenings a week from 7 till 9. Instruction will be given in Qualitative and Quantitative Analysis, and in the Preparation of Chemical Products. Fees:—(Two Terms) Two Evenings, 25s.; One Evening, 15s. (One Term) Two Evenings, 15s.; One Evening, 10s. 6d.

University College, Bristol, has been approved by the Council of the Institute of Chemistry as a College at which all the subjects required for the admission of Associates to the Institute are taught.

The Calendar of the College, price 1s. (post-free, 1s. 4d.), containing detailed information of the various Courses, may be obtained on application to the Secretary.

MERCHANT VENTURERS' TECHNICAL COLLEGE, BRISTOL.

CHEMISTRY AND METALLURGY.

Professor—J. Wertheimer, B.Sc., B.A., F.I.C., F.C.S.

Lecturers—G. P. Darnell-Smith, B.Sc., A.I.C., F.C.S.; H. A. M. Borland, A.R.C.S.

Demonstrators—R. Tutton; F. B. Gatehouse; W. H. Collier (of the University of London)

Assistant in Chemical Laboratory—P. J. F. Pearson.

The Laboratories of this Department are open during the Term on every week-day as a School of Practical Chemistry, Analysis, and Assaying, to Students of either sex. They include the Junior Laboratory (for forty Students working at one time), Senior Laboratory, Gas Analysis Room, Balance Room, Combustion Room, Store Rooms, and Metallurgical Laboratory. Each Student works independently, and receives individual instruction and help.

The training in this Department is intended to fit Students to become Professional Chemists, to prepare them for Medical or Pharmaceutical examinations in Chemistry, to instruct them in the branches of Applied Chemistry required in Manufactures and Agriculture, and to train them in practical Metallurgical processes, especially Assaying.

Special facilities will be given to Students wishing to undertake research work approved by the Principal; and the Courses provide completely for the Preliminary Scientific Examination (M.B.), and for the Examinations in Chemistry for the B.Sc. degree of the University of London.

The three years' course at this College in the subjects of Theoretical and Practical Chemistry, Theoretical and Practical Physics, and Elementary Mathematics, is accepted by the Council of the Institute of Chemistry as

satisfactory evidence of the training of candidates for the Associateship of the Institute, provided the details in each case be certified by the Professors.

Certificates of Proficiency in Assaying will be given to Students who complete a Course of not less than two years' work in the Metallurgical Department, and pass satisfactorily the College examinations in the Theory and Practice of Assaying.

The Laboratories are open every week-day from 9 till 12 noon, and from 1.30 to 4.30 p.m., except Saturday, when they close at noon.

The Annual Fee of £10 10s. secures (in addition to the use of either or both Laboratories) permission to attend the Lectures in Physics and Mathematics as well as Chemistry, and to use the Physical Laboratory and the Engineering Workshop.

The College Evening Classes, available to persons of either sex, will commence on Monday, September 24th.

Further detailed information may be obtained from the College Prospectus, price 6d.

UNIVERSITY OF BIRMINGHAM.

Professor—Percy F. Frankland, Ph.D., B.Sc., F.R.S.

Assistant Lecturer—C. F. Baker, Ph.D., B.Sc.

Demonstrator—W. R. Innes, Ph.D., M.Sc.

Lecturer on Metallurgy—Godfrey Melland, F.I.C.

The Session will be opened on October 2nd, 1900.

Lecture Courses.

A. This part of the course is arranged (1) to give a full exposition of the general principles of Chemical Science, (2) for the systematic study of the properties of the more important elements and their compounds, and (3) to indicate the chief applications of Chemistry in the Arts and Manufactures. Five hours weekly during the Winter and Spring terms. At least one of the above meetings of the class in each week will be devoted to tutorial work. Attendance at this tutorial class is compulsory, as is the performance of the weekly exercises set by the Professor. Mondays to Fridays inclusive, at 9.30 to 10.30 a.m. Fee, £5 5s.

B. This part of the course includes an introduction to the study of Organic Chemistry, with a description of the properties, relations, and methods of preparation of the more important groups of Carbon Compounds. Three hours weekly during the Summer term. Mondays, Wednesdays, and Fridays, at 9.30 to 10.30 a.m. Fee, £1 11s. 6d.

Advanced Organic Chemistry.—This course extends over two years, and is divided into two parts:—(a) Carbon Compounds of the Fatty Series; (b) Aromatic and other Cyclic Compounds. Only one of these parts will be taken in each year. The class meets twice weekly by arrangement during the Winter and Spring terms. Fee, £2 2s.

General and Physical Chemistry.—This course will deal in outline with various Chemical subjects. The class meets once weekly by arrangement during the three terms. Fee, £1 11s. 6d.

A further Course in Advanced Organic Chemistry will deal with one of the above parts of the Course. The class meets two hours weekly by arrangement during the Winter and Spring terms. Fee, £2 2s.

A further Course on General and Physical Chemistry on special subjects attracting attention at the time. Fee, £1 11s. 6d.

Practical Chemistry.

This subject embraces three Courses. The Laboratory will be open daily from 9.30 to 5, except on Saturdays, when it will be closed at 1 p.m. Fees—

	All day.	Three hours per day.	Three hours per day; five days a week.	Three hours per day; three days a week.
	Guineas.	Guineas.	Guineas.	Guineas.
One Term ..	7	4½	4	2½
Two Terms ..	13	8½	7½	5
Three Terms	18	12	11	6½

A Course of short demonstrations and exercises is

given by the Professor or one of his Assistants once a week. All first-year Students are required to attend, unless exempted for special reasons by the Professor. No Fee.

Metallurgy.

Three Courses of Ten Lectures will be given on the Principles and Practice of Metallurgy. Fee, 10s. 6d. for each of the first two courses, and for each of the two sections of the third course. A more advanced course of about sixty lectures upon selected subjects is also given.

There is a separate laboratory for metallurgical students in which provision is made for instruction in assaying, &c.

Scholarships.

Priestley Scholarships.—Three Open Scholarships in Chemistry of the value of about £96 each are awarded annually in September.

Bowen Scholarship.—One Open Scholarship in Metallurgy of the value of about £96 is awarded annually in September.

For particulars apply to the Registrar.

Excursions.

During previous Sessions permission has been obtained to visit some of the great factories in or near Birmingham, in which chemical and metallurgical industries are carried on. Students have thus had most valuable opportunities of gaining a practical acquaintance with some branches of Applied Science. The privilege thus courteously granted by several manufacturers will, it is hoped, be enjoyed in every future Session. The excursions will be conducted by the Professor or Lecturers.

BRADFORD MUNICIPAL TECHNICAL COLLEGE.

CHEMISTRY AND DYING DEPARTMENT.

Head of Department—W. M. Gardner.

Lecturers in Chemistry—W. F. Sutherland, Ph.D. (Geneva), *Chemiste-Diplômé de Zürich*; and S. F. S.ell.

Lecturer in Dyeing—A. B. Knaggs, F.C.S.

Lecturer in Metallurgy—(Vacant).

Lecturer on Botany, Biology, and Pharmacy—W. West, F.L.S., Ex. Pres. Yorkshire Naturalists' Union.

The following courses of instruction are provided:—

I. *General Chemistry Course*, extending over three years, including Lectures in Inorganic, Organic, and Technological Chemistry, Principles of Analysis, Technical Analysis, Chemical Philosophy, Crystallography, Fuels, Lighting and Ventilation, Physics, Mathematics, Mechanics, with Laboratory work in Chemistry, Physics, Bacteriology, and Microscopy.

II. *Chemistry and Dyeing*, extending over three years. Includes most of the above subjects, along with Lectures and practical work in Dyeing, Colour matching, &c.

III. *Chemical Engineering*. Three years' course, preparing Students for positions in Chemical Works, Sewage Works, &c.

IV. *Dyeing*. Extending over one or two years.

V. *Textile and Dyeing*. Arranged for those Students who desire to study the two subjects simultaneously.

VI. *General Pharmaceutical Course*. Prepares for the Minor and Major Pharmaceutical Examinations. Each extends over two years on three half-days per week, and includes Chemistry and Physics, Botany, Biology, Materia Medica, Pharmacy, and Dispensing.

ROYAL AGRICULTURAL COLLEGE, CIRENCESTER.

CHEMICAL DEPARTMENT.

Professor—Prof. E. Kinch, F.C.S., F.I.C.

Assistant—W. James.

Systematic courses of Lectures are given on the various branches of Chemistry in its relation to Agriculture, illustrated by experiments, and by the collections in the College Museum. They comprise the laws of Chemical Combination and the general Chemistry of mineral bodies, and of the more frequently occurring bodies of organic origin, with the relationships of their leading

groups; and, finally, the applications to practical operations of the Chemistry of the atmosphere, of soils and manures, of vegetation, of stock feeding, and of the processes and products of the dairy.

In the Laboratory practical instruction is given in the construction and use of apparatus and in Chemical manipulation and analysis, both qualitative and quantitative. After studying the simple operations and the properties of the commonly occurring substances, the Students are taught to analyse a series of compounds, and apply the knowledge thus obtained to the analysis of manures, soils, waters, feeding stuffs, dairy products, and other substances met with in the ordinary course of Agricultural practice. Chemico-agricultural researches are undertaken by the senior Students under the direction of the Professor and his Assistants.

VICTORIA UNIVERSITY. THE YORKSHIRE COLLEGE, LEEDS.

Professor of Chemistry—Arthur Smithells, B.Sc. Lond., F.I.C.

Lecturer in Organic Chemistry—Julius B. Cohen, Ph.D., F.I.C.

Assistant Lecturer and Demonstrator—T. S. Patterson, Ph.D.

Demonstrators—J. McCrae, Ph.D., and H. M. Dawson, B.Sc.

The Session begins October, 1900.

Lecture Courses.

1. General Course of Chemistry.—Monday, Wednesday, and Friday, at 11.30 a.m. Fee for the Course, £4 4s.
2. Inorganic Chemistry.—Honours Course, Metals. Monday, Wednesday, and Friday, at 9.30 a.m. Fee, £3 13s. 6d.
3. Inorganic Chemistry.—Honours Course, Non-metals. Tuesday and Thursday at 9.30 a.m. Fee, £3 13s. 6d.
4. Organic Chemistry.—Tuesday, Thursday, and Saturday at 12 noon. Fee £3 13s. 6d.
5. Organic Chemistry Honours Course.—Wednesday and Friday at 12 noon. Fee, £2 12s. 6d.
6. Theoretical Chemistry.—Advanced Course. Tuesdays and Thursdays at 9.30 a.m. Fee, £2 12s. 6d.
7. Chemistry for Teachers.—Wednesdays from 2.30 to 5.30 in the first and second terms. Fee, £4 4s.

Laboratory Courses.

The College Laboratory will be open daily from 9 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it will close at 1 p.m.

Fees for the Session—Students working six days per week, £21; five, £18 18s.; four, £16 16s.; three, £13 13s.

Class in Practical Chemistry, Saturday mornings, from 9.30 to 12.30. Fee £1 11s. 6d.

Practical Course in Sanitary Chemistry.—Tuesdays and Thursdays from 2 to 5 p.m., from January to March. Fee, £5 5s.

Dyeing Department.

Professor—J. J. Hummel, F.I.C.

Lecturer and Research Assistant—A. G. Perkin, F.R.S.E.

Assistant Lecturer—R. B. Brown.

This Course extends over a period of three years, and is intended for those who wish to obtain a full scientific and practical education in the art of dyeing. It is suitable for those who purpose in the future to take any part in the direction of the operations of dyeing or printing of textile fabrics, e.g., the sons of manufacturers, calico printers, managers, master dyers, &c.

Leather Industries Department.

Professor—H. R. Procter, F.I.C.

Assistant Lecturer and Demonstrator—Andrew Turnbull, Ph.D.

The full Courses, which extend over a period of either two or three years, are suitable to all who intend to become Technical Chemists in the Leather Industry, or managers of important works, and are recommended to

sons of tanners. The Courses include instruction in chemistry, a modern language, leather manufacture, and practical work in the Leather Industries Laboratory and Dye-house.

Agricultural Department.

Professor—J. Seton, B.Sc.

Lecturer in Agricultural Chemistry—H. Ingle, F.I.C.

The full Course occupies two years, and includes instruction in chemistry, physics, mathematics, geology, botany, forestry, engineering and surveying, and the principles of agriculture, as well as practical work in the various laboratories and out-door agriculture.

Research Students are admitted to the College Laboratories on reduced terms.

Several valuable Scholarships are at the disposal of the College, viz., the Salt, Akroyd, Brown, Emsley, Craven, Leeds City Council, and Clothworkers' Scholarships, and one of the 1851 Exhibition Scholarships. The West Riding County Council Scholarships are tenable at the Yorkshire College.

UNIVERSITY COLLEGE, LIVERPOOL.

Professor—J. Campbell Brown, D.Sc.

Lecturer on Organic Chemistry—C. A. Kohn, B.Sc., Ph.D.

Lecturer on Metallurgy—T. L. Bailey, Ph.D.

Demonstrators and Assistant Lecturers—T. L. Bailey, Ph.D., W. B. Davidson, M.A., D.Sc., Ph.D., C. A. Kohn, B.Sc., Ph.D., and A. W. Titherley, M.Sc., Ph.D.

Assistant—H. H. Froyseil.

The Session commences October 4th.

Entrance Scholarship Examination takes place early in May each year.

The Classes meet the requirements of candidates for the Ordinary B.Sc. Degree, for Chemistry Honours, or for the M.Sc. or D.Sc. Degree in Victoria University; for Degrees in Medicine of Victoria, London, and Edinburgh; for the Pharmaceutical Diplomas; for a special Technological Certificate of University College; and for those studying Chemistry as a preparation for professional, technical, or commercial life. The Classes qualify for the Fellowship of the Institute of Chemistry of Great Britain and Ireland, and other Examination Boards.

Lecture Courses.

General Elementary Course on the principal non-metallic elements and the most important metals, the principles of Chemical Philosophy, and an introductory sketch of Organic Chemistry. Three Terms. Fee, £4.

Engineer's Course of Lectures with Practical Class. Two Terms. Fee, including Practical class, £4.

Pharmacy Courses: Junior, £3; Senior, £3.

Dental Course, Lectures and Practical. Fee, £5 5s.

Course A.—Non-metals. Fee, £3.

Course B.—Metals. Fee, £3.

Course C.—Organic Chemistry. Fee, £3.

Course H.—Special Organic Subjects. Fee, £2.

Course D.—Physical Chemistry. Fee, £2.

Course E.—History of Chemistry and of the Development of Modern Chemical Philosophy. Three Terms. Fee, £2.

Courses F.—Applied Chemistry and Metallurgy: Lectures on Technology are given in connection with Laboratory work at hours to be arranged. The subjects are varied in different years. (1) Alkali and Allied Manufactures. (2) General Principles of Metallurgy. (3) Iron, Steel, and Aluminium. (4) Copper, Lead, Silver, and Gold, and other Metals. (5) Distillation of Coal and Tar Industries. (6) Fuel and Gas. (7) Chemistry Applied to Sanitation. (8) Technical Gas Analysis. (9) Electrochemical work. Three terms. Fee, each course £1 10s.

Practical Classes.

(1) Junior. (2) Intermediate: Qualitative Analysis of Inorganic Substances and of some of the more common Organic Substances; preparation of Inorganic Compounds. (3) Revision Class. (4) Senior: Practical Or-

ganic. (5) Practical Exercises on Technology, Pharmaceutical Chemistry, Sanitary subjects, Examination of Water and Air, of Animal Secretions, Urinary Deposits, Calculi, and Poisons. (6) Quantitative Class.

Chemical Laboratory.

The Chemical Laboratories provide accommodation for every kind of chemical and metallurgical work and research.

The William Gossage Laboratory, opened in 1897, consists of a large and well-fitted general Laboratory for advanced Students, a new gas analysis room, an additional lecture-room for Metallurgy and other classes, and an addition to the Research Laboratory. New stores for students' apparatus and chemicals have also been built and placed in charge of a skilled dealer.

Students desirous of gaining a thorough theoretical and practical acquaintance with Technical Chemistry, or who intend to adopt Chemical work as a profession, must devote three or four years to special study, for which a full curriculum is provided.

TABLE OF FEES.

Per Week.	One Term, Three Months.	Three Terms, One Session.
One day	£4	£7
Two days	5 10s.	10
Three days	7	13
Four days	9	16 10s.
Whole week	10 10s.	21

Pharmaceutical Course (see special syllabus).

Technological Curriculum.

Preliminary Year.—Chemistry, the Elementary Course. Practical Classes 1 and 2. Mathematics, or Mechanics, Elementary Engineering, Drawing, and Design (in this or one of the following years). German. Or, the Victoria Preliminary Course and Examination may be taken.

First Year.—Chemistry—Courses A and B; Chemical Laboratory three days per week; Technological Chemistry, Course F. Physics, with laboratory work, one day per week. Mathematics (intermediate). German. Engineering, First Year Course, Autumn and Lent Terms. Intermediate B.Sc. Examination may be passed.

Second Year.—Chemistry, Lecture Course C, on Organic Chemistry, Lecture Course D or E, Technological Chemistry, Course F. Chemical Laboratory, four days per week. Engineering, Mathematics, or Physics (Advanced). The Final Examination for the Victoria B.Sc., or the Intermediate Examination of the Institute of Chemistry, may be taken.

Third Year.—In the third year the student should begin to specialise. The following are possible alternatives:—1. Courses D, E, and F; Course H. Any other Courses omitted in a previous year. Laboratory, five days per week. The Degree of B.Sc. in the Honours School of Chemistry may be taken. 2. Metallurgical Classes. Metallurgical Laboratory, two days per week. Chemical Laboratory, Gas Analysis, and Electro-chemistry, two days per week. Physical and Electrotechnical Laboratory, one day per week. 3. Courses D, E, and F. Physics (Senior Course). Chemical Laboratory, four days per week. Physical Laboratory, one day per week. 4. Courses D and F, and a course on Chemistry applied to manufacturing operations. Chemical Laboratory, three or four days per week. Engineering Laboratory and Electrotechnical Laboratory, one day per week.

Students may finally choose a special subject either of research or of applied Chemistry. The Final Examination for the Associateship of the Institute of Chemistry may be taken after 1, 2, or 3. Those who have taken the Ordinary Degree of B.Sc. may pass the Victoria M.Sc. Examination in any subsequent year.

The Sheridan Muspratt Chemical Scholarship of £50 per annum, tenable for two years, will be competed for on December 6, 7, and 8, 1900, on an Examination in subjects which are included in the first two and a half years of the above curriculum. Candidates should send in their names to the Principal not later than November 13.

Other Scholarships, Entrance Scholarships, and Free Studentships are also available to Students.

Evening Classes.

Classes, including laboratory work, will be held during the winter.

The Prospectus containing full particulars may be obtained from the Registrar, University College, Liverpool.

DURHAM COLLEGE OF SCIENCE, NEWCASTLE-ON-TYNE.

Professor of Chemistry—P. Phillips Bedson, M.A., D.Sc., F.I.C., F.C.S.

Lecturer in Chemistry—Saville Shaw, M.Sc., F.C.S.

Lecturer in Agricultural Chemistry—S. Hoare Collins, F.I.C., F.C.S.

Assistant Lecturers and Demonstrators—F. C. Garrett, M.Sc., F.C.S., and J. A. Smythe, B.Sc., Ph.D.

1. **General Course.**—This Course of Lectures will extend over the three terms of the Session, and is intended to serve as an introduction to the Science. The Lectures will be of an elementary character, and whilst framed to meet the requirements of First Year Students will also be serviceable to such as intend pursuing Chemistry in its various applications in the arts and manufactures, as, for instance, Brewing, Metallurgy, the Manufacture of Soda, Soap, Glass, &c. The subjects treated will include an exposition of the Principles of Chemistry, and a description of the preparation and properties of the chief Elementary Substances, both metallic and non-metallic, and their more important native and artificial compounds. A section of this Course will be devoted to an outline of Organic Chemistry. The class will meet on Mondays, Wednesdays, and Fridays, at 11 a.m., and will commence on Monday, October 8th. Fee, £3 10s. for the Session.

Second Year Course.—The Lectures for the second year students consist of a course of Lectures on Organic Chemistry, extending from October to March, and a course of Lectures on Inorganic Chemistry from March to the end of the Session. The Class will meet on Tuesdays and Thursdays at 11 a.m., and Fridays at 3 p.m., and will commence on October 9th. Fee for Session, £3 10s.; for Inorganic alone £1 10s.; and for Organic alone £3.

Advanced Classes will be formed for the study of Inorganic, Organic, and Theoretical Chemistry. Fee for the course, £3 10s.

A Lecture Course in Analytical Chemistry will be given on Mondays, at 3 p.m.

Metallurgy and Assaying.—Lecturer, Saville Shaw, M.Sc., F.C.S. A Metallurgical Laboratory is provided, in which instruction is given in the ordinary processes of Dry Assaying, and in the preparation and analysis of Alloys, &c. Fees as for Chemical Laboratory.

Agricultural Chemistry.—The instruction in this branch of Chemistry will consist of a series of Lectures and of special practical work in the Chemical Laboratory. Students will be expected to have a knowledge of Elementary Chemistry, such as may be obtained by attending the General Course.

The Lecture Course in Agricultural Chemistry is arranged for two days a week throughout the Session. Fee, £3 10s.

Practical Chemistry.—The Laboratory is open from 10 a.m. to 1 p.m., and from 2 to 5 p.m., except on Saturdays, when it closes at 1 p.m. **Laboratory Fees.**—Students working two days, £2 10s. per term, £6 per session; one day per week, £1 10s. per term, £3 10s. per session.

Courses of Study.—Students will be divided into two classes:—(1) Regular, or Matriculated Students, who are also Members of the University of Durham; and (2) Non-Matriculated Students. Regular Students will be required to follow such a course of study in the subject professed in the College as will enable them to pass the Examinations for the title of Associate in Science of the University of Durham. Non-Matriculated Students

will attend such classes as they may select. Every candidate for admission as a matriculated student must pass an examination on entrance, in reading, writing from dictation, English or Latin Grammar, arithmetic (including decimals), and geography. Registered students in medicine are exempted from this examination, or students who produce a certificate of having passed either of the three following examinations:—

1. The first examination for the D. degree of B. Litt.
2. Durham Examination for certificate of proficiency in General Education, held in March and September.
3. Durham Examination for Students in Arts in their first year, or any examination of a similar nature that may be accepted by the Council.

Associateship in Science.—Every candidate for the Associateship in Science will be required to satisfy the examiners in—Mathematics, Physics, Chemistry, and either Geology or Natural History—in an examination to be held at the end of the candidate's first year. Associates in Science are admissible one year after obtaining the title of Associate to examination for the degree of Bachelor of Science of the University of Durham.

Exhibitions.—Three Exhibitions of the value of £25, £15, and £10 respectively will be awarded in October next to Candidates desirous of attending the first year course of study in the College.

The examination will be held at the College, and will commence on Tuesday, October 2nd.

Evening Lectures.—Courses of Evening Lectures will be given, with a Practical Class for Laboratory instruction.

Two Exhibitions of £15 each will be awarded at the next examination of "Persons not members of the University," which will be held at Durham in March next.

Several other valuable Scholarships are available for students, including the Johnston Chemical Scholarship of the value of £60 for one year.

OWENS COLLEGE, VICTORIA UNIVERSITY, MANCHESTER.

Professor and Director of the Chemical Laboratory.—Harold B. Dixon, M.A., F.R.S.

Professor of Organic Chemistry.—W. H. Perkin, Ph.D., F.R.S.

Demonstrators and Assistant Lecturers.—George H. Bailey, D.Sc., Ph.D.; W. A. Bone, D.Sc.; P. J. Hartog, B.Sc.; E. J. Russell, B.Sc.; D. L. Chapman, B.A.; and H. C. H. Carpenter, B.A.

Demonstrator in Organic Chemistry.—W. T. Lawrence, B.A., Ph.D.

Lecturer in Technical Organic Chemistry.—Jocelyn F. Thorpe, Ph.D.

Assistant Lecturer in Metallurgy.—Dr. Bone.

The Session begins on October 4, 1900.

The instruction is given by means of Experimental Lectures and Tutorial Classes. The Chemical Classes form part of the Courses for Chemistry in the University.

Chemistry Lecture Courses.

General Chemistry Course.—Tuesdays, Thursdays, and Saturdays, at 9.30, during the two Winter Terms.

Introduction to Organic Chemistry.—Wednesdays and Fridays, at 9.30, during Lent Term.

These courses are intended for Medical Students and others beginning the study of chemistry.

First Year Honours Course.—Mondays, Wednesdays, and Fridays, 11.30 a.m., during the two Winter Terms. The Non-Metals.

Second Year Honours Course.—Tuesdays, Thursdays, Saturdays, 11.30 a.m., during the two Winter Terms. The Metals.

Third Year Honours Course.—At times to be arranged. Physical Chemistry.

Organic Chemistry (General).—Mondays and Fridays, 9.30, during two Winter Terms.

Organic Chemistry (Advanced).—Tuesdays and Thursdays, 9.30, during the two Winter Terms.

History of Chemistry and Chemical Philosophy.—Wednesdays, 9.30, during the Session.

METALLURGY.—**Lectures:** The Metallurgy of Copper, Lead, Silver, Gold, and the Metallurgy of Iron and Steel will be given in alternate years. **Practical:** The Metallurgical Laboratory will be open to students every day.

ELECTRO CHEMISTRY.—In connection with the new John Hopkinson Memorial Laboratory, rooms have been fitted for the study of Electricity in its applications to Chemistry. Courses of lectures and laboratory instruction will be given to Advanced Chemical Students in the coming Session. **Lecturer.**—Mr. R. S. Hutton, B.Sc.

The Chemical Laboratories are open daily from 9.30 a.m. to 4.30 p.m., except on Saturdays, when they are closed at 12.30 p.m.

Courses for B.Sc. Degree.—To qualify for the B.Sc. Degree of the Victoria University, Students have to attend a prescribed course of study extending over three years, and to pass the Preliminary Examination of the University either on entering or at the end of a year's Course.

The Honours Course of Chemistry is as follows:—
First year: First year Honours Lectures; Mathematics (3 hours a week); Physics (3 hours a week); a Language (3 hours a week); Chemical Laboratory (3 days per week).
Second year: Second year Honours Lectures; General Organic Lectures; Applied Chemistry Lectures; Physics Laboratory (1 day per week); Chemical Laboratory (3 days per week).
Third year: Third year Honours Lectures; Honours Organic Lectures; History of Chemistry Lectures; Chemical Laboratory (5 days per week).

The following awards are made to successful Students in the Honours Examination:—A University Scholarship of £50; a Mercer Scholarship of £25. A University Fellowship of £150 is awarded annually among the Graduates in Science for the encouragement of Research. Among the College Scholarships open to Chemical Students are the Dalton Chemical Scholarship, £50 per annum for two years; the 1851 Exhibition Scholarship; the Levisstein Exhibition; &c.

Applied Chemistry.

First Course.—Sulphuric Acid and Alkali Manufactures. General Principles of Chemical Engineering.

Second Course.—The Chemistry of Fuel. The Manufacture of Illuminating Gas and Gaseous Fuel.

Third Course.—Natural and Artificial Dye-stuffs, and the Principles of Dyeing and Printing.

Certificates in Applied Chemistry.

The course extends over a period of three years, and comprises systematic instruction by means of lectures and practical work in the laboratories.

Before admission to the first year's course students are required to give such evidence of elementary knowledge of Mathematics and Chemistry as shall be considered satisfactory by the Senate.

The first year's course is the same for all students working for the certificate.

In the second and third years a choice may be made between Inorganic and Organic Chemistry. By this division of the subject a student wishing to apply himself specially to the inorganic side of the science, may attend during his second year the Honours course in Metals, and courses on Geology or Mineralogy, and during his third year, courses on Metallurgy and on Geology or Mineralogy; while a student wishing to apply himself specially to the organic side of the science, may attend during his second and third years the Courses on Organic Chemistry, and courses on the Coal Tar Colours and on Dyeing and Printing.

Part of the Laboratory practice in the second and third years will consist in the examination and analysis of raw materials, products from chemical works, &c., in connection with the special courses of lectures on Applied Chemistry. In the Chemistry and Physical laboratories the practical work in the second year will be arranged in accordance with the branch of Chemistry selected by the candidate,

In the third year the student, if sufficiently advanced, will be set to work on some analytical process or problem in Applied Chemistry, under the direction of the teaching staff.

UNIVERSITY COLLEGE, NOTTINGHAM.

DEPARTMENTS OF CHEMISTRY AND METALLURGY.

Professor of Chemistry—F. Stanley Kipping, Ph.D., D.Sc., F.I.C., F.R.S.

Demonstrators of Chemistry—J. J. Sudborough, D.Sc., Ph.D., F.I.C., R. M. Caven, B.Sc., F.I.C., and G. D. Lander, D.Sc.

The Classes of the College are open to students of both sexes above sixteen years of age.

The Session commences on October 8th. Evening Classes begin September 24th.

Lecture Courses.—The Chemistry Day Lectures extend over three years. In the first year a student enters for the course on Elementary Inorganic Chemistry. In his second year he attends Lectures on both Inorganic and Organic Chemistry. In his third year he attends courses on Advanced Organic Chemistry, Physical Chemistry, and Advanced Inorganic Chemistry.

The fees for the Day Lectures and Classes are as follows: First year, two Lectures per week, 15s. per term. Second year, three Lectures per week, 22s. 6d. per term. Third year, four Lectures per week, 30s. per term.

Demonstrations and Lectures on Analytical Chemistry will be given in the day and evening, and should be attended by all students.

A Chemical Calculation Class is also held. Fee per Term, 5s.

Students may qualify themselves by attendance at these lectures and classes for the Examinations of the Universities of London, Cambridge, or Oxford, and for the Medical Examinations of the Royal College of Surgeons and of the Universities of Cambridge and Edinburgh: they may also obtain instruction in Chemistry for technical or other purposes, and can enter for a full Chemical Engineering Curriculum. Special attention is given to the requirements of candidates for the Associateship of the Institute of Chemistry.

Practical Chemistry and Metallurgy.—The Chemical and Metallurgical laboratories are open every day from 9 to 5, except on Saturday, when the hours are from 9 to 1; also on Tuesday and Thursday evenings from 7 to 9. Each Student works independently of other Students at a course recommended by the Professor. Instruction is given in general Chemical Manipulation, in Qualitative and Quantitative Analysis, and in the methods of Original Chemical Investigation and Research; Students are also enabled to work out the applications of Chemistry to Pharmacy, Metallurgy, Dyeing, Agriculture, Brewing, Iron and Steel, Tanning, and other Manufacturing Processes. Fees for day students: For one term, £7; for the session, £18; for six hours weekly 40s., and 5s. extra for each additional hour per week. For evening students, 10s. for two hours per week, three hours 15s., four hours 20s., per term.

Research Work.—Students or others wishing to undertake research work in pure or Applied Chemistry will be afforded every facility for doing so and may be admitted at reduced fees. The Laboratories are fully equipped with apparatus and chemicals necessary for such work.

Courses of Technical Chemistry Lectures are also given on Engineering, Dyeing and Bleaching, Brewing, Plumbering, Bread-making, Gas Manufacture, and on other processes of applied Chemistry.

Pharmaceutical Students can at all times work in the Chemical Laboratory, taking work suitable for the preparation for the Minor and Major Examinations. Special lectures will also be given in Chemistry, Materia Medica, and other Pharmaceutical subjects.

Evening Classes.—Evening Lectures and Laboratory instruction will be given in Pure and Applied Chemistry, and the laboratories are open for practical work on Tues-

day and Thursday evenings from 7 to 9. Fee for each Lecture Course, 5s.; for each Laboratory Course, 10s.

Full information concerning all College Classes is given in the College Prospectus, price one penny.

UNIVERSITY COLLEGE, SHEFFIELD.

Professor of Chemistry—W. Carleton Williams, B.Sc., F.C.S.

Lecturer—G. Young, Ph.D., F.R.S.E.

Demonstrator—T. Slater Price, D.Sc.

The Session will commence on October 5th.

Beginners' Course.—Inorganic Chemistry: Tuesday from 10 to 11 a.m. Fee, £1 11s. 6d.

Matriculation Course.—Friday 10 to 11. Fee, £1 11s. 6d.

Intermediate Course.—Inorganic Chemistry. Monday and Thursday from 10 to 11 a.m. £2 12s. 6d.

Organic Chemistry.—Elementary: Saturdays, 10 to 11; fee, £1 11s. 6d. Honours: Tuesdays and Thursdays, 12 to 1; fee, £2 12s. 6d. Advanced: Thursdays, 4 to 5; fee, £1 11s. 6d. Special Course: Hours to be arranged; £1 11s.

Physical Chemistry.—Tuesday, 11 to 12. Fee, £1 11s. 6d.

Chemical Philosophy.—Thursday, 11 to 12. Fee, £1 11s. 6d.

Short Courses of Lectures are also given by L. T. O'Shea on the Chemistry of Coal Mining.

A Course of Lectures is arranged for Medical Students, with a special class in Qualitative Analysis.

Laboratory.—Working hours to be arranged between Professor and Students.

Sessional Fees for Day Students:—Six hours per week, £5 5s.; Nine, £7; Twelve, £8 8s.; Eighteen, £11 5s.; Twenty-four, £14; Thirty-two, £17.

Day Students may not enter for less than six hours a week. Students joining the Laboratory at Christmas will be charged two-thirds and at Easter one-third of the Fees for the whole Session.

Fees for short periods (working thirty-two hours per week):—For one month, £3 3s.; two months, £5 5s.

A course of Elementary Practical Physics and Practical Chemistry, which will meet the requirements of candidates for the Diploma of Public Health and for the Licentiate of Sanitary Science, will be held during the Michaelmas and Lent terms. Fee £5 5s.

An arrangement has been entered into with the Science and Art Department, South Kensington, which will enable Science Teachers to work in the Chemical Laboratory for three, six, or twelve hours a week on payment of one-quarter of the usual fee, the Department being willing to pay the remainder under certain conditions, of which full information may be obtained on application to the Registrar.

Evening Classes.—Lectures, Wednesday, 8 to 9. Laboratory instruction, Wednesday, 6 to 9, and another series to be arranged if desired. Sessional Fee, one evening per week, £1 10s.; two, 50s.; or Lecture Class and Laboratory, on Wednesday evening, £1 10s. Fee for one term, 17s. 6d.

UNIVERSITY COLLEGE, DUNDEE.

UNIVERSITY OF ST. ANDREWS.

Professor of Chemistry—James Walker, Ph.D., D.Sc., F.R.S.

Assistant Lecturers—J. S. Lumsden, Ph.D., D.Sc., and J. K. Wood, M.Sc.

Lecture Assistant and Laboratory Steward—J. Foggie, F.C.S.

The Winter Session begins on October 10th, and ends on March 20th. The Summer Session extends from the middle of April to the end of June.

The First Year's Lecture Course on Systematic Chemistry is given daily during the Winter Session, and embraces the Elements of Inorganic and of Organic Chemistry.

Advanced Courses, of about fifty lectures each, will be given during the year as follows:—

Organic Chemistry; Inorganic Chemistry, including the more important technological applications; Theo-

retical and Physical Chemistry; Bleaching and Dyeing, including the Chemistry of the Textile Fibres.

Practical Instruction in all of the above branches will be given in the Laboratories and Dye-house. Special facilities are afforded to Research Students.

UNIVERSITY OF ABERDEEN.

CHEMISTRY.

Professor—Francis R. Japp, LL.D., F.R.S.

Demonstrators—W. B. Davidson, M.A., D.Sc., and W. Maitland, B.Sc.

I. General Lecture Course (100 Lectures).—Daily during the Winter Session. The subjects treated of include (1) The Laws of Chemical Combination and the General Principles of Chemistry; (2) Non-metallic and Metallic Elements, and their Compounds; (3) Organic Chemistry; (4) Applications of Chemistry to the Arts and Manufactures. Fees, for first attendance, £3 3s.; for subsequent attendance, £2 2s. A Tutorial Class (without fee), conducted by the Senior Demonstrator, is held in connection with this course.

II. Special Lecture Course on Organic Chemistry (50 Lectures).—Daily during the Summer Session. Fee, £2 2s.

III. Practical Course for Medical Students.—This course, which occupies five hours a week during the Summer Session—in all fifty hours—is devoted to practice in Elementary Qualitative Analysis.

IV. Chemical Laboratory.—The Laboratory is open to Students daily from 9 a.m. to 5 p.m. Each Student on entering is allowed to arrange his hours of work so as to suit his own convenience, but must adhere to these hours when once fixed. The Laboratory instruction includes: (1) General experiments; (2) Preparations; (3) Qualitative analysis; (4) Quantitative analysis: gravimetric, volumetric, and gasometric. The fees depend upon the number of hours taken, and vary from £3 3s. to £6 6s. a session. A certain number of free places are available, on the recommendation of the Professor, and subject to the approval of the Senatus, for research students.

In connection with the Laboratory work, two Lectures a week on Advanced Analysis and on Physical Chemistry are delivered (without extra fee) by the Senior Demonstrator.

Agricultural Chemistry.

Lecturer—James Hendrick, B.Sc., F.I.C.

This course is given during the Winter Session, and includes both Lectures and Laboratory work. The Lectures deal with the chemistry of the atmosphere, the soil, manures, and foods. The physiological chemistry of plants and animals, the composition and manurial requirements of crops, and dairy chemistry, are also treated of. The Laboratory work is primarily intended to accompany and illustrate the Lectures. Exercises dealing with the properties and composition of soils, manures, feeding stuffs and waters, and with the impurities and adulterations of these, occupy much of the time given to practical work. The fee for the whole course—lectures and practical work—is £3 3s.

UNIVERSITY OF EDINBURGH.

DEPARTMENT OF CHEMISTRY.

Professor—Alex. Crum Brown, M.D., D.Sc., F.R.S.

Lecturers—L. Dobbin, Ph.D., and H. Marshall, D.Sc.

Assistants—W. W. Taylor, M.A., B.Sc., J. P. Longstaff, and James Kerr, B.Sc.

The working terms are—Winter Session, from middle of October to middle of March; Summer Session, from beginning of May to end of July.

Lecture Courses.—During the Winter Session a General Course of Chemistry for medical and science students is given by the Professor. The class meets daily; fee £4 4s. An Advanced Course of twenty-five lectures is also given in the Winter Session; fee, £2 2s. A class on Organic Chemistry is held in summer; fee, £2 2s. There is also a class on Chemical Theory, by Dr. Dobbin; fee £1 1s.; and a class on Mineralogy and Crystallography, by Dr.

Marshall; fee, £2 2s. All these Lectures, except the General Course, are now open to women.

In addition to the above, Lecture Courses are given by the Assistants on some particular branch of Organic and Inorganic Chemistry. These Lectures are free to Laboratory Students.

Tutorial classes are held in connection with the General Course.

Laboratories.—Practical classes for Medical Students meet daily during the latter part of the Winter Session and in the Summer Session. (Fee, £3 3s.) The laboratories for analytical and advanced practical work are open daily from 9.30 till 4.30. (Fees: Whole Day—Winter Session, £10 10s., Oct.-Dec., Jan.-March; or Summer Session, £5 5s. Half Day—Winter Session, £6 6s., Oct.-Dec., Jan.-March; or Summer Session, £3 3s. Preference will be given to students in the above order. Students who are not Matriculated may attend the Chemical Laboratory on payment of the entrance fee of 5s. in addition to the Laboratory fees. Full Courses of instruction are given in Analytical, Practical Organic and Inorganic Chemistry, including Gas Analysis, Metallurgy, and Assaying. Facilities are afforded to advanced students who desire to undertake chemical investigations.

Various prizes and scholarships are attached to the laboratory and general class.

Graduation.—Two Degrees in Pure Science are conferred, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.).

Candidates for Degrees in Science, if not graduates (by examination) in Arts in one of the Universities of the United Kingdom or in a Colonial or Foreign University recognised for the purpose by the University Court, must pass a preliminary examination in (1) English; (2) Latin, Greek, French, or German; (3) Mathematics; (4) One of the languages Latin, Greek, French, German, Italian, not already taken under (2), or Dynamics. In the case of a student whose native language is other than European, the Senatus may, at the Preliminary Examination, accept such language as a substitute for a modern European language. The Senatus may also in such a case accept as an alternative to Latin or Greek any other classical languages, such as Sanscrit or Arabic.

The First B.Sc. Examination embraces Mathematics, or Biology (*i.e.*, Zoology and Botany), Natural Philosophy, and Chemistry. The Final B.Sc. Examination includes any three or more of the following subjects:—Mathematics, Natural Philosophy, Astronomy, Chemistry, Human Anatomy, including Anthropology, Physiology, Geology, including Mineralogy, Zoology, including Comparative Anatomy, and Botany, including Vegetable Physiology. In the Final Examination two written papers are set in each subject professed, the second of a higher standard than the first. Candidates must pass the first section in all, and the second section in at least one, of the subjects professed; the same regulations apply also to the Practical and Oral Examinations. Chemistry in this examination embraces Inorganic, including Mineralogical, Chemistry; Organic Chemistry; Chemical Crystallography; History of Chemistry. In the written papers a choice of questions is allowed, so as to adapt the examination to the various courses of advanced study which candidates may have selected. Practical Examination:—Complex Qualitative Analysis; Inorganic Preparations; Gravimetric and Volumetric Analysis. Each candidate taking the higher standard will also be examined on Organic Preparations, Ultimate Organic Analysis, and one of the following subjects, selected by himself:—Gas Analysis; Assaying; Physico-chemical Measurements.

A candidate for the D.Sc. Degree must submit a thesis on original work done by him. The Thesis must be approved before the candidate is allowed to proceed to Examination. The candidate in Chemistry may be required to pass a searching examination in one of the following branches:—(1) The Chemistry and Chemical Technology of Inorganic Bodies, including Metallurgy;

(2) Organic Chemistry; and to show a thorough practical acquaintance with chemical analysis in all its branches, and with the preparation of pure substances.

HERIOT-WATT COLLEGE, EDINBURGH.

Principal—F. Grant Ogilvie, M.A., B.Sc., F.R.S.E.

Professor—John Gibson, Ph.D., F.R.S.E.

Assistant Professor—Allan W. C. Menzies, M.A., B.Sc.

Demonstrator—Andrew F. King.

The Session begins October 2nd, 1900.

The curriculum of this College comprises both Day and Evening Classes, each department providing the higher general and technical education.

Chemistry.—The first course for day students is a combination of Lectures with Laboratory instruction. In the Lectures some of the more important elements and their compounds are discussed in detail, so as to lead to a knowledge of the general laws of chemical action. Other important elements are treated in less detail, and the relations and classification of the elements generally are broadly indicated. In the Laboratory each student will receive instruction in general chemical manipulation, in accurate weighing, volumetric measurement, and in some of the simpler methods of quantitative analysis. After making a series of simple preparations, he works through a number of experimental exercises illustrating chemical combination, oxidation, reduction, and double decomposition. These exercises are followed by instruction in simple methods of qualitative analysis, especial attention being given to dry way testing and the use of the spectroscope. Students attending a further course may take up the study of systematic analysis, and extend the knowledge they have gained of quantitative analysis by exercises in gravimetric, volumetric, and electrolytic methods. Ultimately they may make a speciality of any branch of the subject which may be most necessary for their future work. Great attention has been paid to the thorough equipment of Advanced Laboratories, and special facilities are given to advanced students who may wish to engage in any class of Research (Inorganic or Organic) whether of a purely chemical or of a technical nature.

The teaching in the Evening Classes is based on the Syllabus of the Science and Art Department, and includes Elementary, Advanced, and Honours Courses in Theoretical and Practical Inorganic and Organic Chemistry.

GLASGOW AND WEST OF SCOTLAND TECHNICAL COLLEGE.

Professor of Chemistry—G. G. Henderson, D.Sc., M.A.

Professor of Technical Chemistry—E. J. Mills, D.Sc., F.R.S.

Professor of Metallurgy—A. Humboldt Sexton, F.C.S., F.R.S.E.

Also, Professors and Lecturers in the other leading branches of Pure and Applied Science and Technology.

The main objects of this College are to afford a suitable education to those who wish to qualify themselves for following an industrial profession or trade, and to train teachers for technical schools. It was founded by an Order in Council, dated 26th November, 1886, according to a scheme framed by the Commissioners appointed under the provisions of the Educational Endowments (Scotland) Act, whereby Anderson's College, the Young Chair of Technical Chemistry in connection with Anderson's College, the College of Science and Arts, Allan's Glen Institution, and the Atkinson Institution were placed under the management of one governing body.

The Diploma of the College is awarded to Day Students who have attended prescribed courses of instruction and passed the necessary examinations. The ordinary courses extend over three years, but arrangements are made for advanced students continuing their studies in special departments.

Complete courses of instruction are provided in both Day and Evening Classes.

Copies of the Calendar for 1900-1901 may be had from Mr. H. F. Stockdale, the Secretary, 38, Bath Street, Glasgow, price by post, 1s. 4d. Prospectuses will be sent free.

UNIVERSITY OF ST. ANDREWS.

UNITED COLLEGE OF ST. LEONARD AND ST. SALVATOR.

Professor of Chemistry—T. Purdie, B.Sc., Ph.D., LL.D., F.R.S.

The Session begins on October 10th. A Competitive Examination, open to intending Students of Arts, Science, and Medicine, for about forty Entrance Bursaries, ranging in value from £40 to £10 each per annum, will be held on September 28th and following days. Twenty-six of these Bursaries are restricted to Men and about fourteen to Women, the latter being intended for women who at the conclusion of their Arts or Science Course will proceed to Medicine. One Scholarship of £80, tenable for one year, will be open for competition to Graduates of Science at the close of Session 1900-1901.

A Hall of Residence is provided for Women Students.

Two Degrees in Science are conferred by the University of St. Andrews, viz., Bachelor of Science (B.Sc.) and Doctor of Science (D.Sc.), and Chemistry is also included in the curriculum for the M.A. Degree; the regulations will be found in the "University Calendar."

Lecture Courses.

Two distinct Courses of Lectures are given, each comprising at least one hundred meetings of the class.

First Year's Course.—This Class meets at 11 o'clock on five days in the week. The introductory lectures treat of the Nature of Chemical Action, the Classification of Substances into Elements and Compounds, the Phenomena of Oxidation, and the Composition of Air and Water. The Laws of Chemical Combination and the Atomic Theory are next discussed, after which the more commonly occurring elements and inorganic compounds are described systematically. Elementary Organic Chemistry is also included in the Course.

The chemistry of manufactures is referred to only cursorily; special attention, on the other hand, is given to those parts of the science which are of general educational value, and as much of the theory of chemistry is introduced as is compatible with elementary treatment. The Lectures are supplemented by a short Course of Laboratory Practice, intended to illustrate the principles of the science.

These courses of instruction are intended to meet the requirements of the Arts' Curriculum; also of candidates for the First B.Sc. Examination, and of students of medicine, so far as Theoretical Chemistry is concerned.

Second Year's Course.—The first part of the Course is devoted to Organic Chemistry, and the second part treats of the General Principles and Theory of Chemistry, and of more advanced Inorganic Chemistry, the instruction in general being such as is required for the Second B.Sc. Examination.

Certificates are awarded on the results of examinations, and the "Forrester Prize" of about £10 is awarded to the best Student of the year.

Fee for the Session, for each Course, £3 3s.

Practical Chemistry.

The Laboratory is open daily from 9 a.m. to 4 p.m., except on Saturdays, when it is closed at 1 p.m. The work pursued in the Laboratory comprises:—

(1) The performance of experiments illustrative of the Principles of Inorganic and Organic Chemistry; (2) Qualitative and Quantitative Analysis; (3) Original Investigation. Each student pursues an independent course of study under the supervision of the Professor or Demonstrator, the nature of the work varying with the proficiency of the student and the particular object he may have in view. Suitable courses of instruction in Practical Chemistry are provided for candidates for

the First and Second B.Sc. Examinations, and for Students of Medicine.

The fees for Practical Chemistry vary according to the number of hours taken weekly. A certain number of working places in the Laboratory will be available without fee for students who are capable of undertaking original investigation.

QUEEN'S COLLEGE, BELFAST.

Professor—E. A. Letts, Ph.D., D.Sc., F.R.U.I., F.R.S.E., &c.

I.—Chemistry.—The lectures are delivered at 3 p.m., on the first five days of each week, and terminate at the end of March. The course is divided into three parts:—(1) Chemical Philosophy; (2) Inorganic Chemistry; (3) Organic Chemistry. Fee, £2.

II.—Practical Chemistry.—In this course the Students are instructed in the general methods of conducting Chemical Analyses. Fee, £3.

III.—Laboratory Pupils.—The Chemical Laboratory is open from November until the end of March, and from May 1st until the third week of July, on the first five days of the week, from 10 a.m. until 4 p.m. Students are admitted as working pupils on payment of a fee of £5 for the first period, or of £3 10s. for the second period (or for a single term).

The new Chemical Laboratories are now open, and are provided with all modern appliances. Special facilities are offered to those engaged in Research work.

Scholarships.—The following College Scholarships are awarded specially in connection with the schools of Chemistry and Physics:—Senior Scholarships in Chemistry alone awarded annually of £40, and tenable for one year. Andrews Studentship in Chemistry and Physics, value about £80 annually, and tenable for two years. 1851 Exhibition Scholarships: One of these Scholarships has hitherto been placed at the disposal of the College, of the annual value of £150, tenable for two or under special conditions for three years.

The following Scholarships, &c., of the Royal University of Ireland are awarded in the same group of subjects (Chemistry and Physics):—At the B.A. Degree examination, First-class Exhibitions of £42 each and Second-class Exhibitions of £21. At the M.A. Degree examination, a Studentship of £100 per annum, tenable for three consecutive years. Also a Junior Fellowship, tenable for four consecutive years, of the annual value of £200.

QUEEN'S COLLEGE, CORK.

Professor—Augustus Edward Dixon, M.D.

Demonstrator—Robert Elliott Doran, F.C.S.

The College Session begins on October 16th, 1900, and ends on June 8th, 1901. The classes are open to male and female students.

Systematic Chemistry.—(1) General course of Inorganic Chemistry, Elementary Organic Chemistry, and Chemical Philosophy.—Fee for each Sessional Course, £2. Each subsequent Course, £1. (2) Advanced Organic Chemistry, and Chemical Philosophy.

Practical Chemistry.—(1) Two ordinary Courses of Practical Chemistry will be held, each of three months' duration; one commencing on January 4th, 1901, and adapted to the requirements of Students proceeding to the Examinations of the Royal University of Ireland; the other ending about the third week in June, and suitable for Medical Students intending to present themselves for the Examinations of other Licensing Bodies. Fee for each Sessional Course, £3. (2) A Course for Pharmaceutical Students will be held in the second and third terms; fee, £5. (3) Special Courses.

The Chemical Laboratory is open daily from 10 to 4 o'clock (except during class hours and on Saturdays) under the Superintendence of the Professor, to Students entering for special courses of qualitative and quantitative analysis; organic chemistry; or for the purpose of original investigation.

QUEEN'S COLLEGE, GALWAY.

Professor—Alfred Senior, Ph.D., M.D., F.I.C.

Demonstrator—W. S. Mills, B.A.

The College Session, 1900-1901, commences October 16 and ends June 8.

Chemistry is studied by attendance at Lectures, by work in the Laboratories, and by the use of the College Library. The Courses in the several faculties are arranged with a view to the requirements of the Royal University of Ireland, but are adapted also to those of other Universities and licensing bodies.

Lecture Courses. Faculty of Arts.—1. Second year's Course, Inorganic and the Elements of General Chemistry. 2. Third year's Course, Advanced Organic Chemistry. 3. Fourth year's Post-Graduate Course, Advanced General Inorganic and Organic Chemistry. **Faculty of Medicine.**—First year's Course, Inorganic and Elementary Organic Chemistry. **School of Engineering.**—First year's Course, Inorganic Chemistry.

Laboratory Courses. Faculty of Arts.—1. Second year's Course, Exercises in Inorganic Qualitative Analysis. 2. Third year's Course, Quantitative Analysis and other experiments to suit the requirements of individual Students. 3. Fourth year's Post-Graduate Course, Advanced Quantitative Analysis, Organic and Inorganic Preparations, and determination of their Physical and Chemical characters. 4. The Laboratories are also open to Students for work in other branches of Chemistry. **Faculty of Medicine.**—1. Second year's Course, Inorganic and Organic Elementary Qualitative Analysis, and the Chemical Examination of Urine. **School of Engineering.**—1. Second year's Course, Inorganic Qualitative Analysis.

A Scholarship in Chemistry is offered for competition each year of the value of £40, and Chemistry enters into the subjects required for numerous Scholarships in Arts, Medicine, and Engineering. The Royal Commission for the Exhibition of 1851 offer a Scholarship of £150 per annum. This Scholarship has just been awarded for the second time to a Chemistry Student of this College.

For Fees, Regulations as to Scholarships, and other particulars apply to the Registrar, from whom the Calendar, published in December, and the Extracts from Calendar, published in advance in July, may be obtained.

ROYAL COLLEGE OF SCIENCE FOR IRELAND, STEPHEN'S GREEN, DUBLIN.

Professor of Chemistry—W. N. Hartley, F.R.S.

Assistant Chemist—J. Holms Pollok, B.Sc., Associate of the Royal College of Science, Dublin.

Demonstrator of Chemistry and Assaying—S. E. Bellanders.

The Royal College of Science for Ireland supplies, as far as practicable, a complete course of instruction in Science applicable to the Industrial Arts, and is intended also to aid in the instruction of teachers for the local Schools of Science.

Diplomas are awarded in the Faculties of Mining, Engineering, and Manufactures, Physics, and Natural Science. If accompanied by a certificate from the Professor of Chemistry, the Diploma of Associate of the Royal College of Science in the Faculty of Manufactures is recognised by the Council of the Institute of Chemistry of Great Britain and Ireland as qualifying candidates for admission to the practical examinations of the Institute.

The instruction in Chemical Science includes (1) General Chemistry; (2) Advanced Chemistry, including Organic Chemistry; (3) Analytical and Experimental Chemistry; (4) Instruction in Chemical Research.

Fees payable by Non-Associate Students:—£2 for each separate Course of Lectures. For Analytical Chemistry and Research—£2 for a special course of one month; £5 for three months; £9 for six months; £12 for the entire session. For Assaying—£5 for three months; £9 for six months; £12 for the entire session.

The following are supplementary courses of instruction:—

- (1) Laboratory Instruction in the Theory of Chemistry.
- (2) An Analytical Course for Students in Engineering.
- (3) A Course of Practical Chemistry for Medical Students.
- (4) The Analysis of Water, Air, Food, and Drugs, intended for the instruction of Public Analysts and Medical Officers of Health.
- (5) Assaying.
- (6) A Course of Chemistry for Pharmaceutical Students.

There are four Royal Scholarships of the value of £50 each yearly, with Free Education, including Laboratory Instruction, tenable for two years; two become vacant each year; they are awarded on the results of their examinations to Associate Students, not being Royal Exhibitioners, who have been a year in the College. There are also nine Royal Exhibitions attached to the College, of the yearly value of £50 each, with Free Education, including Laboratory Instruction, tenable for three years; three become vacant each year, and are competed for at the May Examinations of the Department of Science and Art.

CHEMICAL LECTURES, CLASSES, AND LABORATORY INSTRUCTION.

CITY AND GUILDS OF LONDON INSTITUTE FOR THE ADVANCEMENT OF TECHNICAL EDUCATION.—The operations of the City and Guilds of London Institute are divided broadly into four branches: the educational work of three London Colleges, and of the Technological Examinations. Programmes of the London Colleges may be had on application to the Head Office of the Institute, Gresham College, Basinghall Street, London, E.C., or from the respective Colleges. The Technological Examinations (Examinations Department, Exhibition Road, S.W.), are conducted once every year at various centres throughout the kingdom. Programme, with Syllabus of Subjects, &c., may be obtained of Messrs. Whittaker and Co., Paternoster Square, London, or through any bookseller, price 10d., net. — *City and Guilds Technical College, Exhibition Road.*—Professor of Chemistry, H. E. Armstrong, Ph.D., F.R.S. The object of this Institution is to give to London a College for the higher technical education, in which advanced instruction shall be provided in those kinds of knowledge which bear upon the different branches of productive industry, whether Manufactures or Arts. The main purpose of the instruction given is to practically demonstrate the application of different branches of science to various manufacturing industries. In order that this instruction may be efficiently carried out, the Institution, in addition to the lecture theatres and class rooms, is fitted with laboratories, drawing offices, and workshops; and opportunities are afforded for the prosecution of original research, with the object of the more thorough training of the students, and for the elucidation of the theory of industrial processes. The courses of instruction are arranged to suit the requirements of—1. Persons who are training to become Technical Teachers; 2. Persons who are preparing to enter Engineers' or Architects' offices, or Manufacturing works; 3. Persons who desire to acquaint themselves with the scientific principles underlying the particular branch of industry in which they are engaged. The Matriculation Examinations began on Tuesday, September 18th, and the Winter Session opens on Tuesday, October 2nd. *City and Guilds Technical College, Finsbury.*—Professor of Chemistry, Raphael Meldola, F.R.S. The operations of the Technical College, Finsbury, are divided into two distinct portions: Day Classes for those who are able to devote one, two, or three years to systematic technical education; Evening Classes for those who are engaged in industrial or commercial occupations in the daytime and who desire to receive supplementary instruction in the application of Science and of Art to the trades and manufactures in which they are concerned or employed. Each Professor is assisted by Demonstrators. Besides these there are Lecturers

and Teachers in special subjects. An examination for the admission of Students was held at the College on Tuesday, September 18th. Session begins October 2nd. *South London Technical Art School.*—Classes in Modelling, Design, Drawing and Painting, House Decoration.

CITY OF LONDON COLLEGE, White Street, Moorfields.—Courses of Evening Lectures and Laboratory Practice in Chemistry and Physics, conducted by Mr. I. S. Scarf, F.I.C., F.C.S., assisted by Messrs. H. W. Harrie, F.C.S., and C. A. West, F.C.S., &c. Session commences Oct. 1.

BATTERSEA POLYTECHNIC.—Principal, Mr. Sidney H. Wells, Wh. Sc. Inorganic, Organic, and Technological Chemistry, Mr. John Wilson, M.Sc. (Vit.), assisted by Mr. John L. White, M.Sc., and Mr. B. C. Polkinghorne, B.Sc. Day and Evening Classes in Science and Art subjects.

BIRKBECK LITERARY AND SCIENTIFIC INSTITUTION, BREAM'S BUILDINGS, CHANCERY LANE.—Chemistry Courses will be conducted, commencing September 26, adapted for the Elementary, Advanced, and Honours Examinations of the Science and Art Department, and for the Matriculation, B.Sc., and M.B. Degrees of the London University, by Mr. J. E. Mackenzie, Ph.D., B.Sc.

BOROUGH POLYTECHNIC INSTITUTE, 103, Borough Road (near the Obelisk).—Chemistry: Evening Lectures and Laboratory Work (Session fees, per course, 8s. to 15s.), Dr. F. Mollwo Perkin, assisted by Dr. A. J. Walker. Physics: Evening Lectures and Laboratory Work (Session fees, per course, 8s. to 10s.), Dr. J. Henderson. Special Evening Courses on Oils and Fats, including Soap and Candle Manufacture, and on Painter's Oils, Colours, and Varnishes. Session opens Monday, September 24, 1900.

BRIXTON SCHOOL OF CHEMISTRY AND PHARMACY, 12, Knowle Road, Brixton, London.—Principal, Dr. A. B. Griffiths, F.R.S.E., F.C.S. Lectures on Chemistry, Physics, Botany, &c. The School is fitted with the best appliances, and there are collections of rare chemicals, metals, essential oils, &c. Medals and certificates are awarded to successful students. Practical Chemistry in all branches; and there are courses of Bacteriology, Biological Chemistry, and Research work for advanced pupils. The Laboratories are fitted with every convenience. For particulars apply, in writing, to Dr. Griffiths.

SOUTH WESTERN POLYTECHNIC, Chelsea, S.W.—Principal, Herbert Tomlinson, B.A., F.R.S. Professor of Chemistry, J. B. Coleman, A.R.C.S., F.I.C. Day and Evening Courses in Theoretical and Practical Chemistry and several branches of Applied Chemistry, including Metallurgy, Oils and Colours, and Photography. Special Classes are held for the Matriculation, Intermediate Science, and B.Sc. Examinations of the University of London. Prospectuses of Day and Evening Classes may be obtained from the Secretary, 1d. each, by post 2½d.

THE GOLDSMITHS' INSTITUTE, New Cross, S.E.—Head of the Chemistry Department, Mr. W. J. Pope; Assistants, Mr. S. J. Peachey and others. Lectures and Practical Classes in General Chemistry and Photography, also in Chemistry applied to other industries, are held in the evenings from 7.15 to 10.0, and are open to both sexes. Special attention is paid to Technical Laboratory work and the investigation of manufacturing difficulties.

NORTHERN POLYTECHNIC INSTITUTE, Holloway, N.—Principal, J. T. Dunn, D.Sc. Evening Classes in Theoretical and Practical Chemistry. Assistants, Mr. H. Charles L. Bloxam and Mr. W. H. Watson, A.R.C.S. Prospectus containing detailed information of Chemistry Courses, as well as related Courses in Physics and other departments, may be obtained at the Institute.

NORTHAMPTON INSTITUTE (City Polytechnic), St. John Street Road, E.C.—Principal, R. Mullineux Walmsley, D.Sc., &c. Lecturers in Technical Chemistry: Mr. C. J. Head, F.I.C., &c.; Mr. S. Field, A.R.C.S.; and Mr. J. E. Evans. Day and Evening Classes in Electro-chemistry. Electro-plating, Electro-metallurgy, Electrotyping and Stereotyping, and Gold and Silver Assaying. (For full details see Prospectus).

CARPENTERS' COMPANY TECHNICAL INSTITUTE, Jupp Road, Stratford, E.—Principal, William Ping. F.C.S. Session begins Sept. 24th. Evening Classes in Theoretical and Practical Chemistry, and in many other Departments of Science and Art. Day Technical School.

EAST LONDON TECHNICAL COLLEGE, People's Palace, E.—Chemistry: Professor, J. T. Hewitt, M.A., D.Sc., Ph.D.; Demonstrator, F. G. Pope; Assistants, H. A. Phillips and A. J. Turner. Lectures and Practical Classes are held in the daytime in connection with the three years' course of the Day Technical College. Evening Classes are also held, offering instruction in the courses of the Science and Art Department and for the examinations of the University of London. The Day College opened on Sept. 10. Evening Classes begin Sept. 24.

S. THOMAS CHARTERHOUSE INSTITUTE AND SCHOOLS, Goswell Road, E.C.—President, Rev. Henry Swann, M.A.

UNIVERSITY TUTORIAL COLLEGE, 32, Red Lion Square, Holborn, W.C.—This Institution contains large Laboratories for Chemistry, Physics, Geology, and other subjects. Classes are specialised for Examinations of London University, but students may take up work for any Examination. The special feature of the College is that work goes on all the year round, thus affording students residing in the Country an opportunity of doing practical work during their Vacations.

SOUTH LONDON SCHOOL OF PHARMACY, Lim., 325 and 409, Kennington Road, S.E.—Lectures on Chemistry and Physics, by Dr. John Muter, F.R.S.E., F.I.C., and Mr. J. Thomas, B.Sc. (Lond.), Daily, at 12 noon. Lectures on Botany daily at 11 a.m. by Dr. Muter, and at 2.30 p.m. on Materia Medica and Pharmacy, by Mr. W. J. Mawer, Ph.C. The Students' Laboratory of this Institution is specially designed to accommodate 40 Students. The Technical Laboratory is open daily from 9 till 5, and is fully fitted with all apparatus for teaching the manufacture of drugs and chemicals. Periodical Examinations of the Students are held by Visiting Examiners appointed by the Council of Education, and Medals and Certificates are awarded on the results thereof. Fees: for the first term, 12 guineas; for the second and third terms, 10 guineas; and for the full session 27 guineas, inclusive of all departments. A special laboratory for training Advanced Students in Analytical Chemistry under the care of Mr. A. H. Mitchell Muter, F.I.C., F.C.S.

IMPERIAL COLLEGE OF CHEMISTRY AND PHARMACY, 51, Imperial Buildings, Ludgate Circus.—Mr. F. Davis, B.Sc. An especial course of instruction is given at this College in Pharmacology and Therapeutics, *vs* the Institute of Chemistry Examination.

LONDON COLLEGE OF CHEMISTRY AND PHARMACY.—Principal, Henry Wootton, B.Sc.

METROPOLITAN COLLEGE OF PHARMACY, 100 and 162, Kennington Park Rd.—Principal, W. Watson Will, F.C.S.

THE CLIFTON LABORATORY, Berkeley Square, Bristol.—Principal, E. H. Cook, D.Sc. (Lond.), F.I.C. Students are received either as Private Pupils or Members of a Class. Instruction is given to those requiring to use science or scientific methods in Commercial and Industrial pursuits, or in preparing for Examinations. Students are urged to undertake researches and receive special attention from the teachers. Every effort is made to produce thorough chemists rather than successful examinees.

LEEDS TECHNICAL SCHOOL (in connection with the Leeds Institute of Science, Art, and Literature), Cookridge Street, Leeds.—Head Master, R. E. Barnett, B.Sc. (Lond.), A.R.C.S. Evening Classes are held in 31 subjects of Science and Technology, among which are:—Chemistry, Inorganic and Organic, Theoretical and Practical, by Mr. R. E. Barnett, B.Sc., A.R.C.S., Mr. R. W. Ferguson, A.R.C.S., and Mr. Chas. Walker, B.Sc.; Metallurgy, Theoretical and Practical, and Iron and Steel Manufacture, by Mr. B. A. Burrell, F.I.C., F.C.S.; Magnetism and Electricity, and Practical Physics, by Mr. J. E. Tindall, B.Sc.; Botany, Lectures and Practical Work, by Mr. N. Walker. Fees:—Lectures, from 2s. 6d. per Session;

Laboratory work, from 5s. Systematic courses arranged for Pharmaceutical Students and for those engaged in Chemical Industries at reduced composition fees. Session commenced Sept. 17th. Prospectuses of various Classes free on application to the Secretary, Mr. Arthur Tait. Calendar of the Institute (150 pp.) post free 4d.

LEEDS COLLEGE OF PHARMACY.—Principal, F. Pilkington Sargeant, F.C.S., &c.

THE MUNICIPAL TECHNICAL SCHOOL, Princess Street, Manchester.—At this important Municipal School, with an attendance of upwards of 4300 Students, there are organised Day Courses in Pure Chemistry, with applications to Dyeing, Bleaching, Printing, Brewing, and Metallurgy. In addition there are Evening Courses, not only in Pure Chemistry, but in Metallurgy, Mineralogy, Iron and Steel Manufacture, Brewing, Bleaching, Dyeing, and Printing, Coal Tar Products, Paper Manufacture, Gas Analysis and Gas Volumetric Calculations, Gas Manufacture, Technical and Commercial Analysis, the Chemistry of Oils and Fats, Chemical Engineering, Chemistry for Engineers and Builders, and Photography. The complete Syllabus (6d., by post 9d.) may be obtained on application to Mr. J. H. Reynolds, Director and Secretary, Princess Street, Manchester.

REDRUTH SCHOOL OF MINES, CORNWALL.—Principal, J. Paul de Castro, B.A. (Cantab.), A.I.C., F.C.S., assisted by a staff of Mining Engineers and Mine Surveyors. Courses of Practical and Theoretical instruction are given in Inorganic Chemistry, Assaying, Mineralogy, and Blow-pipe Analysis, for intending Assayers and Mineral Chemists. Shorter Courses are also held in the above subjects to meet the requirements of intending Mining Engineers, and Prospectors. Practical instruction in Mining given at Pednandrea, the School Mine. Syllabus on application to the Registrar.

READING COLLEGE.—Lecturer in Chemistry, C. M. Luxmoore, D.Sc., F.I.C. Assistants, J. L. E. Drugman, Ph.D., and W. J. Monk. Autumn term begins Oct. 3.

TECHNICAL INSTITUTE, SALFORD.—Principal, W. Wilson, M.A. Session begins September 12th. Special attention is directed to the Dyeing and Calico Printing Laboratories. Day and Evening Classes in Science subjects. Also School of Art. Particulars free from Secretary.

STOCKPORT TECHNICAL SCHOOL.—Department of Chemistry and Dyeing.—Principal: Mr. R. J. Brown, M.Sc. A syllabus with full particulars of the courses of instruction, hours, fees, &c., is obtainable on application. Special instruction is given in the Dyeing of Felt, &c., as applied to Hat Manufacture.

WOLVERHAMPTON FREE LIBRARY SCIENCE AND TECHNICAL SCHOOL.—Inorganic and Organic Chemistry, Mr. W. Whitehouse, F.C.S.; Physics, Mr. H. J. Tench; Botany, Mr. Sidney Phillips, Ph.C.; Latin, Mr. F. G. Griffiths, M.P.S. Day Classes in Chemistry and Physics, and Evening Classes in Chemistry, Physics, Botany, French, Latin, &c. Special arrangements are made for the requirements of Pharmaceutical and Medical Students. The School is recognised by the Examining Board of the Royal College of Surgeons and Physicians, as a centre for preparation in Chemistry and Physics. Session commenced Monday, Sept. 17th. For other particulars and programme, apply Mr. J. Elliot, Secretary.

Analyst requires temporary use of Chemical Laboratory in London for Mineral Analyses, &c.—State weekly rental, including use of necessary Apparatus and Chemicals, &c., to "Zero," CHEMICAL NEWS OFFICE, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.

Advertiser requires position as Chemist or Assistant Manager in Works. Six years' experience in Works in England, including entire control, and two years Assayer in mines abroad. Honours Science Graduate, M.Inst.M.E.—Address, E. E., CHEMICAL NEWS OFFICE, 6 & 7, Creed Lane, Ludgate Hill, London.

Situation as Chemist or Assayer required by young man, A.I.C. Three years at College; three years in Public Analyst's Laboratory; and one and a-half years in Works as Assayer. Good references.—Address, A.19, CHEMICAL NEWS OFFICE, 6 & 7, Creed Lane, Ludgate Hill, London, E.C.



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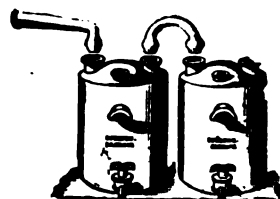
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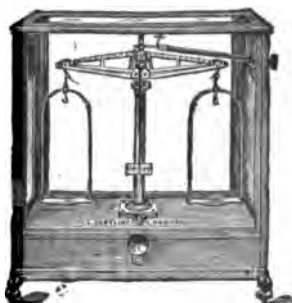
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2131.

ON SOME PROBLEMS CONNECTED WITH ATMOSPHERIC CARBONIC ANHYDRIDE, AND ON A NEW AND ACCURATE METHOD FOR DETERMINING ITS AMOUNT, SUITABLE FOR SCIENTIFIC EXPEDITIONS.*

By Professor LETTS, D.Sc., Ph.D., &c., and R. F. BLAKE,
F.I.C., F.C.S., Queen's College, Belfast.

ATTENTION is drawn to the variations in the amount of atmospheric carbonic anhydride which correspond with at least 10 per cent of the total amount, the causes of which are still to a large extent obscure. In the authors' opinion the subject is an important one, and is worthy of a systematic investigation by a number of skilled observers working in different localities and employing the same method of determination which shall have been proved to give results which do not vary from the true amount by more than three or four parts per million of air. Among the problems relating to atmospheric carbonic anhydride which the authors think are specially deserving of attention are the following:—

1. *Is Schläsing's Theory Correct?*—Do the oceans really act as regulators of the amount of atmospheric carbonic anhydride by the production or dissociation of earthy bicarbonates according as the amount rises above the normal or falls below it? As consequences of this theory latitude should influence the quantity of atmospheric carbonic anhydride, which ought to be lower in polar than in tropical localities, and the great ocean currents should also have an effect as they pass from warmer to colder regions, or the opposite.

2. *The Influence of Day and Night at Sea.*—To account for the increased quantity of atmospheric carbonic anhydride over land surfaces at night, which most of the observers have found, two theories have been advanced: (a) cessation of plant activity in decomposing the gas owing to the absence of light, and (b) the streaming out of ground air from the soil owing to the lowering of temperature.

At sea no such influences can be exerted, but an absorption of atmospheric carbonic anhydride may occur at the surface of the water owing to lowering of temperature, thus reversing the land effect.

3. *The Effects of Atmospheric Precipitates, and especially of Snowfall, which appears to increase the amount of Atmospheric Carbonic Anhydride.*—No reasonable theory has been advanced to account for this curious phenomenon, and it would be interesting to ascertain whether it occurs at sea as well as on land; and the same remark would apply to fog and rain, both of which appear to affect the amount also.

Other supposed causes of variation are worth studying, such as the effects of the seasons, direction and force of the winds, the prevailing type of weather, &c. But those which the authors think most interesting are such as a scientific mission would be under peculiarly favourable conditions for observing, and especially the proposed Antarctic expeditions.

In a memoir of the authors recently published in the *Proceedings of the Royal Dublin Society* (vol. ix., N. S., Part II., No. 15) a modification of Pattenkofer's process

for determining atmospheric carbonic anhydride is described by means of which results of great accuracy may be obtained. Thus, in the final set of test experiments with artificial mixtures of purified air and carbonic anhydride in known volumes, a mean error (in six determinations) of about 1 per cent of the gas was found, corresponding with some four parts per million of air taken.

For use by a scientific expedition it seemed, however, to the authors that a different process is required, in which the operations at the place of observation should be as simple as possible, and of such a nature as to permit of the actual determinations being made at any convenient time later, when the resources of a properly equipped laboratory are available.

The authors have accordingly devised a method which fulfils these conditions, and which is simple and accurate. On the one hand, it resembles Pattenkofer's process in that a relatively small volume of air is examined (about 6 litres), while, on the other, Müntz and Aubin's principle is adopted of absorbing the carbonic anhydride by caustic potash solution and afterwards liberating it by ebullition *in vacuo* with an acid, and measuring its volume in a suitable gas analysis apparatus.

A series of sealed tubes is prepared in the laboratory, each tube containing an accurately measured volume of weak potash solution (the amount of combined carbonic anhydride which such a solution always contains having been ascertained for a given stock).

The only operations which have to be performed at the place of observation are the collection of the air sample in a suitable receiver; the transfer of the contents of one of the sealed tubes to the latter, and, after absorption of the atmospheric carbonic anhydride, their re-transfer, as far as possible, to the same tube, which will be again sealed. The tubes can, of course, be kept for an indefinite period both before and after their contents have been thus treated, and the determination of the absorbed carbonic anhydride made, when convenient, with an aliquot portion of their contents. The experiments made to test the accuracy of the new method were satisfactory. Artificial mixtures of purified air and carbonic anhydride in definite volumes were employed (the two being in about the proportion they occur in ordinary air). Five determinations in such mixtures gave a mean error of 1.3 per cent of the carbonic anhydride taken, equivalent to 4 parts per million of air.

NEW SILICON COMPOUNDS OBTAINED BY THE USE OF THE ELECTRIC FURNACE.*

By CHARLES S. BRADLEY.

THE compounds in question are silicides of calcium, barium, or strontium, from which, by a secondary step, silico-acetylene is obtained.

They have the formula CaSi_2 , BaSi_2 , and SrSi_2 respectively, and are the silicon analogues of the alkaline earth carbides, while the silico-acetylene is the analogue of acetylene having the formula Si_2H_2 , when the carbonates or oxides of the alkaline earths are mixed with silica in the form of ground quartz or sand, in which the relative atomic proportions of the alkaline earth metal to the silicon in the mixture is as 1 is to 2, and sufficient carbon to effect the reduction is added, or when silicates of the alkaline earth metals in which the atomic relation of the earth metal to the silicon is as 1 to 2, are mixed with sufficient carbon to take up the oxygen of the compounds present, and heated in the electric furnace under conditions substantially like those maintained in the manufacture of alkaline earth carbides, silicides of the alkaline earth metals are produced.

* Read before the British Association (Section B), Bradford Meeting, 1900.

* Read before the Chemical Section of the British Association, Bradford Meeting, 1900.

As an example of the process the following reactions for the formation of barium silicide from the barium compounds may be given:—

- (1) $\text{BaCO}_3 + 2\text{SiO}_2 + 7\text{C} = \text{BaSi}_2 + 7\text{CO}$.
- (2) $\text{BaO} + 2\text{SiO}_2 + 5\text{C} = \text{BaSi}_2 + 5\text{CO}$.

Calcium and strontium silicides are formed by exactly similar reactions from similar compounds. They are white or bluish-white substances of metallic appearance, and also resemble aluminum silicide and silicon somewhat in appearance.

They possess a distinctly crystalline fracture, showing plate-like crystals very similar to those seen in the fracture of cast zinc, the crystals being, however, somewhat smaller in size.

They oxidise slowly in the air and more rapidly under the influence of heat, yielding silicon dioxide and the oxide of the alkaline earth metals present. Like the carbides they are decomposed by water, but yield, instead of acetylene, hydrogen in a pure state, which is evolved without explosion, the following being the reaction:—

- (1) $\text{CaSi}_2 + 6\text{H}_2\text{O} = \text{Ca}(\text{OH})_2 + 2\text{SiO}_2 + 10\text{H}$.
- (2) $\text{BaSi}_2 + 6\text{H}_2\text{O} = \text{Ba}(\text{OH})_2 + 2\text{SiO}_2 + 10\text{H}$.
- (3) $\text{SrSi}_2 + 6\text{H}_2\text{O} = \text{Sr}(\text{OH})_2 + 2\text{SiO}_2 + 10\text{H}$.

The calcium compound dissolves slowly in cold water but more rapidly in hot water; the barium compound decomposes rapidly in both cold and hot water. The strontium compound dissolves more rapidly in water than the calcium, but not so rapidly as the barium compound.

It will be noticed, by considering the equations 1, 2, and 3, that all of these compounds evolve large volumes of hydrogen:—

- 1 lb. CaSi_2 producing 0.104 lb. or 18.73 cu. ft. hydrogen at 0° C. and 760 m.m.
- 1 lb. BaSi_2 producing 0.051 lb. or 9.15 cu. ft. hydrogen at 0° C. and 760 m.m.
- 1 lb. SrSi_2 producing 0.069 lb. or 12.36 cu. ft. hydrogen at 0° C. and 760 m.m.

Calcium silicide, when treated with dilute acids, either the oxy-acids or the hydrogen acids, gives rise to the formation of a new compound which has the formula Si_2H_2 , and is therefore the silicon analogue of acetylene, C_2H_2 , and must be called silico-acetylene, since it bears the same relation to silico-methane (silicon hydride), SiH_4 , as acetylene bears to methane, CH_4 , the reaction being $\text{CaSi}_2 + 2\text{HCl} = \text{CaCl}_2 + \text{Si}_2\text{H}_2$. Silico-acetylene is a yellow crystalline compound, and differs in properties from the compound SiH_2 which Ogier obtained by sparking SiH_4 , which was unstable and exploded when subjected to a shock, Si_2H_2 being stable or non-explosive at ordinary temperatures.

When treated with 20 per cent solution of caustic soda or potash, (Si_2H_2) yields hydrogen according to the following equation:—



Heated in air this compound, Si_2H_2 , oxidises rapidly, giving $2\text{SiO}_2\text{H}_2\text{O}$, and when heated in a closed tube it breaks down into amorphous silicon and free hydrogen. Strontium silicide, when treated with a strong acid, does not produce silico-acetylene with the same facility, while the barium compound when so treated produces a mixture of gaseous compounds and free hydrogen. These silicides can be produced at low cost where electric power is cheap, are very powerful reducing agents, and it is hoped will find large use in the dye industries. Some experiments have been tried on molten steel carrying phosphorus and sulphur, and the requisite quantity of silicide of barium or calcium completely removed these impurities as well as all oxygen present. These compounds were discovered and examined by Mr. Charles B. Jacobs, of New York.

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A LARGE field of applied electrical engineering has been developed of late years in connection with mining work. Not only has electricity been brought to the aid of the miner, working underground, but, after the raw materials are brought to the surface, in the various processes of the purification of the metal, it holds away. As a means of producing motive power, electricity holds its own against its rivals—steam, gas, water-power, &c.—chiefly by virtue of its economy, adaptability, and flexibility; as a means of producing chemical effects, electricity stands unrivalled, and for this reason one of the most important of modern industries is that of applied electro-chemistry. The equivalent of thousands of horse-powers of electrical plant are already in use for the separation and purification of various metals.

The Boston and Montana Copper and Silver Mining Company of Great Falls, Montana, have one of the largest and best equipped copper refining works in the world. The power required for these works is obtained from the river, the power-house being placed on the bank about 3 miles above the town of Great Falls. Here is installed a twin water-wheel, working under a head of 50 feet of water. This wheel develops 2000 h. p. at 130 revolutions per minute, and drives two large electrical generators.

The generators, which are direct coupled, one at each end of the water-wheel shaft, are of Westinghouse construction, and are each capable of yielding continuously 1250 k. w. at 220 volts pressure. They run on this full load continuously, seven days a week, day and night. The field magnets of the machines are divided vertically, and each half slides on horizontal guides, thus leaving the armature accessible without the necessity of disturbing any of the machine bearings. This arrangement is very convenient where head-room is limited and cranes are not at hand for lifting heavy weights. The coupling between the water-wheel and each generator consists of two steel discs, each with projecting studs or pins. A strong leather ring is placed over each corresponding pair of studs, one on each disc. The brush-holders are carried on a ring supported on the field magnets; by this arrangement the commutator of the machine is left as clean as possible for cleaning and inspection.

The lead of all the brushes is adjusted simultaneously by a hand-wheel on a worm shaft gearing on to the rim of the supporting ring.

The generators are independently excited by means of two small Westinghouse generators yielding current at a 125 volts pressure.

The switchboard for the control of this plant is 7 feet 6 inches in height and 10 feet long. It is split up into panels, four carrying the regulating gear of the main generators, and one the regulating gear for the exciter. The whole is of Westinghouse construction; the four large single-pole switches for the main generator circuits have a carrying capacity of 6000 amperes each.

The works containing the copper refining plant is situated on the summit of a hill about half a mile away from the power-station. There were two courses to choose from as to how the electrical energy should be conveyed from the generating works to the refining plant. It meant either raising the pressure of the current to a high value and installing a light transmission line, in which case large converting plant would have been necessary at each end; or it meant keeping the initial low pressure, installing a conductor of large cross-section between the two places, and not requiring any converter plant whatever. The latter arrangement was adopted, and the mains between the power-house and the refining works are exceptionally large. They are of copper strip or plate, 12 inches wide and 2 inches thick, supported the whole way on wooden scaffolding at a height of about 12 feet above the ground.

The copper ore, as it comes from the earth, is ground up and concentrated. The rough metal is then melted

up, cast into pigs, and sent on the works we are dealing with—the refining works.

The refining process is carried out in a number of electrolytic baths. The pigs of metal are each about 2 feet square and 2 inches thick, with a pair of projecting lugs at one end. They are hung on the baths by these lugs from conducting brackets. Thin copper sheets are hung between the pigs and from the cathodes of the bath. The pigs are thus split up, and the refined copper is deposited on the thin sheets. The refuse from the copper pigs falls to the bottom of the bath. It consists chiefly of lead, silver, and gold, and is stated to be worth about £520 a ton; the value of the quantities of gold, silver, and lead extracted from these residues being sufficient to pay for the whole electrolytic process. Work in this refining process is carried on quite continuously, and thus the electrical plant is put severely to the test, and is found to acquit itself perfectly.

The works of the Anaconda Copper Mining Company have also been equipped with electrical plant. Here are installed nine Westinghouse electrolytic generators. Three of these are driven by belt from steam-engines, run at 260 revolutions per minute, and are each capable of producing 3600 ampères at 75 volts pressure. Four other similar-sized machines are direct coupled to steam-engines, and the remaining two are larger-sized generators, 4800 ampères at 75 volts, also direct coupled to steam-engines. A peculiar feature of these electrical generators is that each of them is provided with two commutators, one at each end of the armature, the object being to collect the large currents without undue heating at the points of commutation. In addition to the prevention of commutator heating, the weight is more evenly distributed on the machine bearings, and better brush contacts are secured. The rest of the construction of these machines is practically similar to those supplied to the Boston and Montana Company.

These works are lighted by electricity generated by a Westinghouse alternator driven by water-wheels.

The published annual report of the Boston and Montana Company for 1898 tends to show that the business of copper refining is a profitable one. Their sales of copper, gold, and silver amounted to about £1,500,000, and after payment of all expenses the amount applicable to dividends was £700,000.

It is therefore little to be wondered at that many new works are being opened in the various parts of the United States for the development of this business.

THE UNIVERSAL EUDIOMETER.

By G. H. WOOLLATT.

This instrument is designed to give accurate readings of the temperature, pressure, and volume of a gas through a considerable range of all or any of these factors. It is therefore specially suitable for the demonstration of the laws of Boyle and Charles, and for the analysis or synthesis of gases.

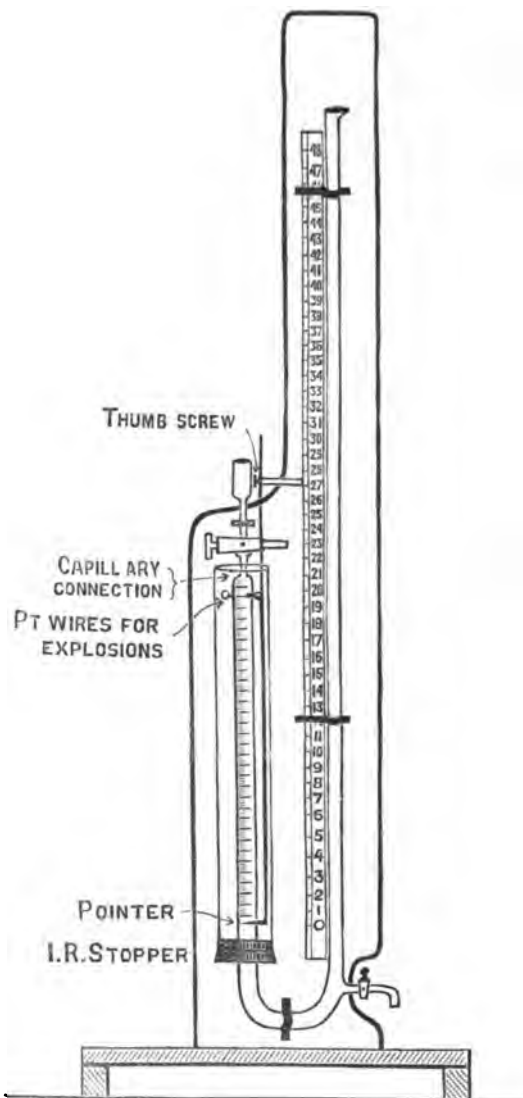
It consists essentially of a U-tube, one limb being jacketed and closed at the top by a three-way cock, the other extending open to a height somewhat over 1 metre. The short limb is provided with explosion terminals, is accurately graduated in volumes, and the shape is such as to allow the whole contents of the tube to assume the temperature of the liquid in the jacket, while still giving easy access to the three-way cock. In order to use a minimum amount of mercury, the longer limb is made of smaller diameter than the short one.

A movable scale, graduated in tenths of inches or centimetres, slides up and down by the side of the longer limb outside the jacket, and a pointer rod attached to the scale slides inside the jacket close to the graduations, the pointer corresponding exactly to the zero of the scale.

In measuring volumes and temperatures, the readings are taken as usual.

(a). *For Pressures Exceeding Atmospheric.*—The pointer is brought to the mercury level in the eudiometer tube, and the height at which the mercury stands—as shown on the scale—in the longer limb, is added to barometric pressure.

(b). *Less than Atmospheric.*—The pointer rod is raised by means of a thumb screw until it corresponds with 10,



20, 30, or 40 c.m. (as is most convenient) on the scale. This is shown by lines engraved on the pointer rod. The pointer is brought to the mercury level in the eudiometer tube as before, and the difference of levels (as read from the scale) subtracted from barometric pressure.

The accompanying sketch will show the type of apparatus. It has proved of the greatest help in lecture demonstrations, giving accurate results with very little trouble, and over a wide range of conditions.

SYNTHETIC INDIGO.*

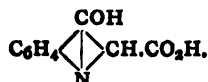
By EDWARD S. JOHNSON.

(Concluded from p. 129).

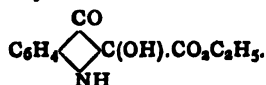
II. THE CONSTITUTION OF INDIGO (*continued*).

Of the remaining atoms of the indigo molecule, two each of hydrogen and oxygen, and the question as to the former's combination with nitrogen, still require consideration and disposition. They have been fixed in their relationships to the others by researches which discovered the constitution of indoxyllic acid and its derivatives. Of the latter, the oximes or isonitroso-compounds, and of isatine as well, are of first importance. The study of the reactions of indoxyl with the aldehydes and ketone acids, with isatine and ethylpseudoisatine threw additional light upon the subject, and incidentally discovered the chromophoric group of indigo; they furthermore strongly corroborated the evidence adduced by the investigation of the nitrous acid derivatives of indoxyl and isatine.

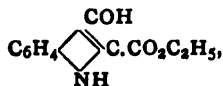
If in indoxyllic acid (ester), which is transformed by gentle oxidation into indigo, the position of the oxygen be established and the question as to the existence of the imido-group in its molecule be answered, the positions of the extra-benzene hydrogens and the two oxygen atoms in indigo are likewise as definitely determined. The constitution of indoxyllic acid (ester) as assumed by Baeyer immediately after his earlier investigation of the subject was—



It will be recalled that the acid was formed from indoxyllic ethylester, which in turn was obtained by reduction of isatogenic ethylester. As already pointed out in another connection, indoxyllic ethylester becomes by gentle oxidation indoxanthinic ethylester. In consideration of all pertinent circumstances, the formula of this substance can only be—



The presence of the imido-group, which is an essential feature, is demonstrated by the formation of a *nitrosamine*. Reduction of indoxanthinic ester forms indoxyllic ester in which the presence of the imido-group must likewise be assumed, it being highly improbable that in the reduction the imido-hydrogen of the indoxanthinic ester should be removed (Baeyer, II., "Ueber d. Verb. d. Indigogruppe, V.," *Ber. Deut. Chem. Gesell.*, xv., 775). The formula of indoxyllic ester should therefore be—



and not as first published. The presence of the imido-groups and a corresponding position of the oxygen atoms in indigo may be presumed with a like degree of certainty.

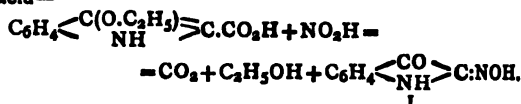
In his extended research upon the nitrous acid derivatives of indoxyl and isatine, Baeyer found that these bodies react according to two formulæ, those representing them in the free state, and other isomeric combinations designated pseudo-forms and corresponding to certain derivatives conceived as generated from the first by the migration of an hydrogen atom. The pseudoforms exist only as derivatives. The formulæ below will explain the distinction:—



Among the derivatives of these combinations, *isonitroso-pseudoindoxyl* or *pseudoisatine- α -oxime*,—



is of great interest from its still more direct bearing upon the question of the position of the oxygen atoms and the existence of the imido-groups. The compound is produced by the action of nitrous acid upon ethylindoxyllic acid—



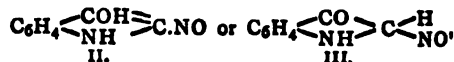
Water may be regarded as being added, forming—



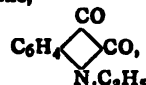
The splitting off of alcohol and carbonic anhydride would leave—



which with nitrous acid gives the isonitroso-compound. That a body of the given composition (I.) and not of two other possible configurations results,—



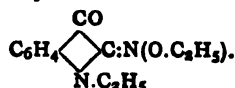
was demonstrated by the behaviour of the ethylation product obtained by the use of one molecule of ethyliodide and sodium alcoholate. In the first place, the derivative by reduction and subsequent oxidation yields isatine, and not ethyl(pseudo)isatine,—



showing thus the ethyl-group not to have substituted hydrogen in the imido-group, but, if Formula I. be correct, in the hydroxyl of the isonitroso-group. Formula II. would require substitution in the hydroxyl and a compound unstable to boiling acid; the substance, on the contrary, resists decomposition by boiling concentrated hydrochloric acid. If III. be assumed, the ethylation must have taken place on the carbon atom binding the nitroso-group—a supposition inconsistent with the easy transformation, by the successive processes of reduction and oxidation, into isatine. Of the three discussed expressions for the substance,—



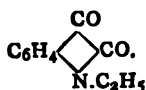
is most in accord with its behaviour. By further action of the sodium alcoholate and ethyl iodide (one mol. each), one (Baeyer, IV., "Ueber d. Verbindungen d. Indigogruppe, V.," *Ber. Deut. Chem. Gesell.*, xvi., 2188) more ethyl-group was introduced. The resulting compound, its mode of formation, its analysis, its resistance to alkalis and even boiling acids considered, must be constituted as indicated by the formula below:—



This compound, *ethylpseudoisatine- α -ethyloxime*, was converted by Baeyer by very gentle reduction into *diethyl-*

* *Proceedings of Engineers' Society of Western Pennsylvania*, xvi., No. 3.

indigo. Oxidation of this compound produces ethyl-pseudoisatine,—

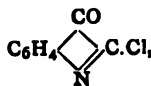


This reaction together with the process of formation furnishes indubitable evidence that the ethyl-groups of diethylindigo are bound by nitrogen. Confirmative of these observations and in analogy with the first, *pseudoisatine- α -oxime*,—

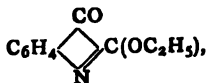


by the same agency produces *indigo*. By these remarkable experiments, the existence of the imido-groups in the indigo molecule was established, and the position of the oxygen atoms given beyond reasonable doubt.

As confirming the conclusion with reference to the disposition of the oxygen atoms, Baeyer directs attention to the fact that the moderate reduction of isatine chloride,—



and ethylisatine,—



both bodies of the indigo group, which contain oxygen combined with the carbon atom next to the benzene-ring, and only such, produce indigo.

From still another standpoint, the question as to the constitution of indigo has been illuminated. Baeyer observed that mixtures of aqueous solutions of *indoxyl* with certain aldehydes give rise to stable red precipitates, accompanied by the formation of one molecule of water. *p*-Nitrobenzaldehyde, to take a simple instance, undergoes a similar reaction. Indoxyl and nitrous acid likewise produce a yellowish red precipitate, also with the separation of one molecule of water. These phenomena, it was concluded, were of themselves sufficient evidence for the assumption of similarity of constitution. The conclusion, however, became unquestionable when it was discovered that both (the nitrous acid derivative after substitution of the hydrogen of the oxime-group by ethyl) form with sodium alcoholate blue salts whose solutions in chloroform show the spectrum of indigo. The nitrous acid derivative is the already discussed pseudoisatine- α -oxime,—



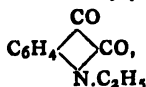
By analogy, the compound from *p*-nitrobenzaldehyde would be—



Such bodies Baeyer denominated *indogenides*, and the dyad atomic complex—



contained by them all, *indogen*. Its substitution in place of oxygen in the simple aldehydes (acetaldehyde, benzaldehyde), and others by application in aqueous solution, produces yellow crystalline unstable substances. When derived from other more complicated aldehydes (*p*-nitrobenzaldehyde, terephthalic aldehyde), and also benzaldehyde (with indoxyl in dry condition), red stable precipitates are obtained. With *ethylpseudoisatine*,—



an indogenide remarkably resembling indigo in properties

is formed. Finally, the synthesis of indigo from pseudoisatine- α -oxime, whose constitution has had detailed consideration, discovers indigo itself as *di*-indogen,—



Throughout these reactions, the chromophoric character of nitrogen is fully exhibited. Colorific properties are generated by its substitution in intrinsically colourless substances, and these increasingly as the complexity of the substituted compound increases. At the beginning of the series there is the yellow and unstable indogenide from acetaldehyde, and, in its culmination, the exceedingly stable and intensely blue indigo.

Thus, by cumulative evidence gathered from divergent lines of investigation with wonderful skill, the proposition around which these unique researches centre is left upon an abundantly substantiated experimental basis.

III. SYNTHETIC INDIGO IN ITS RELATION TO THE VEGETABLE PRODUCT.

Vegetable indigo is formed as a glucoside in the cells of plants of the genus *indigofera*, growing in India, Africa, and Central America, in general, in a tropical climate. The Indian crops are of greatest importance both in point of quality and quantity. The seed of the plant is there sown in rudely tilled soil and harvested after a few months, just before the blooming time. Two crops are grown each year. The colouring matter is secreted mainly by the leaves. To prepare it, the green plants are steeped in earthenware or wooden vats in water and allowed to ferment during nine to fifteen hours, according to the temperature. A yellow fluid is formed and run off into a vat, placed conveniently below the first. It is here agitated, to facilitate access of air for oxidation, mechanically or by hand. The fluid soon begins to change colour to green and later blue, with the gradual separation of the colouring matter as a blue precipitate. After clearing, the supernatant fluid is removed and the precipitate transferred to a third vessel, where it is boiled with water for several hours to still fermentation and to some extent purify it. Collection upon filter-cloths, expressing the adhering water, forming by cutting into cubes or lumps, drying, and packing complete the preparation for the market.

Vegetable indigo is not the nearly pure substance represented by its rival, synthetic indigo, but besides contains indirubine, indigo-brown, glutinous substance, ash, and other impurities derived from the mode of preparation. In the adulterated colour, chalk, lime, ashes, and the like are found. The percentage of indigo may vary from 20 to 90 per cent.

The Indian indigo crop for each year from 1879 to the present time is given in the accompanying table.*

The yield for 1898 was 124,580 maunds or 11,012,872 lbs., in value at the average price (*Journ. Soc. Dyers and Colourists*, Indigo Chart, Oct., 1898) of 64 c. for that year, nearly 7,050,000 dols. Allowing that the Indian production is five-eighths of the total, and that of other growers proportionally good at the price given, the value of the total yield for 1898 would be about 11,275,000 dols. (Otto N. Witt, *Prometheus*, xi., 371, estimates the value of the world's production of indigo at 15,000,000 dollars).

In competition for this prize, the leading representative of Germany's chemical industry, the great Badische Anilin u. Sodafabrik, has entered with synthetic indigo by processes described or modifications of them. It may not be amiss, particularly at the present juncture in its development, to pause for a moment's consideration of the magnitude of this industrial giant (the data are from 1896). Its products are chiefly the coal-tar colours of

* *Amer. Wool and Cotton Reporter*, Oct. 12, 1899, p. 1209. By the courtesy of Messrs. Kuttroff, Pickhardt, and Co., New York, my attention has been directed to this and other statistical information here reproduced. I am further indebted to the same firm for samples of artificial indigo used in illustration of the lecture.

Year.	Factory maunds.
1879	73,128
1880	136,200
1881	135,405
1882	150,278
1883	159,388
1884	166,507
1885	108,692
1886	131,261
1887	130,825
1888	132,354
1889	144,718
1890	100,733
1891	150,506
1892	87,231
1893	116,329
1894	160,400
1895	162,200
1896	158,800
1897	110,212
1898	124,580

every class, embracing the aniline, alizarine, azo, resorcin, and gallic acid colouring matters. It further manufactures every variety of auxiliary and intermediate product consumed in the above preparations, including all products of the acid, soda, and chlorine industry. It was founded in 1865, and is now situated in Ludwigshafen on the Rhine, opposite Mannheim, one of the most influential commercial centres of Southern Germany, and has branch works in France and Russia. The main concern at Ludwigshafen employs 100 scientifically trained chemists, 30 engineers, and a commercial and accountant force of 230 men. In 1896, including overseers and labourers, the employees of the works numbered about 4800. The factories occupy 220 acres, of which 60 acres are covered by buildings of the works, and 492 dwellings for labourers and 78 for officials. Within its limits there are 21 miles of railway. The consumption of coal yearly is 190,000 tons. For the generation of steam for the heating of apparatus and running 190 steam engines of 6500 horsepower, there are 83 boilers. The water-works of the establishment provide annually 2640 million gallons of water, an ice plant supplies over 13,000 tons of ice, and the gas-works generate nearly 340 million cubic feet of gas. For electric lighting, there are four dynamo-machines of 1000 horse-power supplying current for 6400 incandescent lights and 470 arc lights. There are nearly three acres of shops for repairs of every description and the construction of special apparatus.

This establishment owns the principal and promising patents relating to artificial indigo and intermediate products required in its preparation. In connection with investigators already named, it has, since Baeyer's *o*-nitrocinamic acid synthesis, almost incessantly worked upon the problem of rendering methods discussed practicable. Experiments, in the main, have been directed towards a cheapening of the necessary materials and intermediate products. After a period of twenty years, difficulties have been so far overcome that synthetic indigo is an established article of trade, making its appearance somewhat generally first in 1897. Already it is making itself felt by a material reduction in the price of indigo. In April, 1880, the year of Baeyer's first promising synthesis, medium quality of Bengal indigo sold for 1.87 dols. per pound; in December, 1898, one year after the introduction of synthetic indigo, at 87 c. Good medium Kurpah brought 90 c. in January, 1890, and only 62 c. in December, 1898, in the London market. In September of last year the synthetic preparation was quoted at 38 c. in New York (*Oil, Paint, and Drug Reporter*, September, 1899).

When Wöhler discovered a synthesis for urea, nearly seventy five years ago, the tilling of a fertile field in chemical research was begun. One by one a long list of compounds, in Wöhler's time generally regarded as ex-

clusively within the creative power of the vital force resident in the cells of vegetable and animal organisms, has become a proud trophy of searching investigation.

The knowledge acquired by these triumphs over mystery as connected with the animal organism has acted with beneficence upon the development of higher physiology and the science of medicine. By the synthesis of alizarine and indigo, the vegetable world has been invaded with results already prominently presented in the first instance, and those of similar significance are now confidently regarded as fast approaching a brilliant industrial climax for synthetic indigo.

ON THE SYNTHESIS OF COMPLEX EQUATIONS.

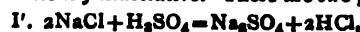
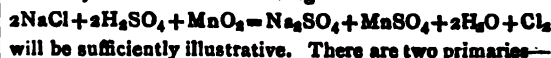
By WILLIAM ACKROYD, F.I.C., Public Analyst for Halifax.

THE regular building up of complex chemical equations receives no attention in text-books. The student is usually left to arrive at an expression of his facts by a series of shots or approximations, or else to commit already constructed equations to memory, to be recalled as required.

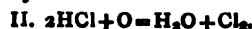
Their systematic synthesis appears to the writer of sufficient importance for him to send to the *CHEMICAL NEWS* the following brief notes of the method he has followed for some years with advanced students and to advocate its wider use. It is briefly:—

- (1) To select the necessary primary equation or equations;
- (2) To work out the naturally following secondary or tertiary, &c.;
- (3) To cancel common quantities; and
- (4) To add up the results for the final complex equation.

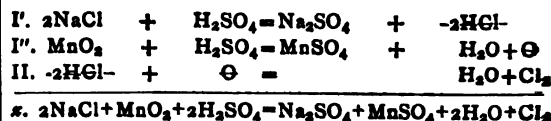
The synthesis of the following—



and one secondary—



The arrangement of quantities, cancelling of those common to both sides, and the final addition to get the complex equation are as follows:—

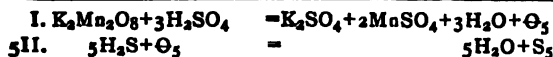


Where the inner nature of this reaction is mentioned, it is usual to suppose that the secondary equation refers to the action of HCl on MnO₂ to liberate the necessary chlorine (*vide* "Reynolds's Experimental Chemistry," 1890, Part II., p. 72), but synthesis on this basis yields contradictory results.

The action of acid (H₂SO₄) permanganate of potash on various reducing agents, such as H₂S, H₂SO₃, H₂O₂, FeSO₄, and KI, may be taken as examples of the system which the method lends itself to; they have one primary equation in common, and are all of the following type:—



The primary equation is the one representing the oxygen available on the action of the acid, and as it is five times that required in the secondary the latter has to be taken five times in the addition, as follows:—



It is unnecessary to proceed with further examples, as the chief interest of the subject to the reader will be in his own application of the synthesis to various complex equations.

ON COMMERCIAL LIQUID CARBONIC ACID.

By J. C. A. SIMON THOMAS.

THE widely different prices charged by various manufacturers of liquid carbonic acid in steel cylinders, for use in ice-making machines on board men-of-war in the Dutch navy, have led me to examine into the purity of the carbonic acid coming from different sources. I have only been able to find two papers on this subject; one a very short one by Fleck (*Vierteljahrsschr. Nahrungs und Genussmittel*, 1899, iv., 78), in which the author states that in using ordinary hydrochloric acid which contains arsenic, for preparing the carbonic acid used in breweries, this latter may contain chloride of arsenic; the other, a preliminary communication from Grünhut (*Chem. Zeitung*, 1895, 505 and 555), in which the author gives a method of analysis. Grünhut did not find any gaseous impurities in the three samples he examined. On the other hand, in every case he noticed the presence of organic and reducing matters by passing the gas through sulphuric acid, which was turned brown, and through either acid or alkaline permanganate, which was decolourised after a few hours. These impurities come from the residue which he found in the cylinders after having emptied them of gas. This residue consisted of a thick brown liquid, which contained principally glycerin and oxide of iron. He is of the opinion that the glycerin comes from the compressor, and that in the presence of water and carbonic acid it has dissolved the iron. The author does not give any information with regard to the manner in which the carbonic acid he examined was prepared.

Commercial liquid carbonic acid is prepared in four different manners (*Zeit. für Angewandte Chemie*, 1898, p. 1193): from natural carbonic acid gas which is found in certain volcanic regions; from carbonates of lime or magnesia; by the combustion of coke, according to Osouf's method; and from the carbonic acid which is formed during fermentation.

The different cylinders of carbonic acid which I have had pass through my hands were from the first three sources. There does not yet appear to be any commercial carbonic acid from fermentation, in Europe at any rate, although I have been told that it is manufactured by Guinness and Co., Dublin.

The general course of the experiments was as follows:—

The first thing to determine was whether the gas which came off contained any gaseous impurities non-absorbable by a solution of soda, and, if such was the case, the nature of these impurities. The gas was then passed through a solution of iodine in iodide of potassium for several hours, for the detection of sulphurous acid, through a solution of acetate of lead for the detection of sulphuretted hydrogen, and, according to Grünhut's method, through concentrated sulphuric acid, and acid and basic solutions of permanganate, to show whether organic impurities were present or not.

I may as well say at once that I have never found either sulphurous or sulphydric acid, nor organic matter, to be present.

After the qualitative, we proceed with the quantitative analysis. The gas is passed into a vessel over mercury, and the carbonic acid absorbed with a solution of soda. The water is estimated as in the preliminary examination, by passing the gas through chloride of calcium or potash

tubes; thus the amounts of carbonic acid and water are determined simultaneously, and the water calculated from these quantities.

The gaseous impurities are carbonic oxide and air, and as, apparently, these gases occur principally in the first portions of carbonic acid that come off, the estimation by means of soda is repeated after the cylinder has been partially emptied. The amount of carbonic acid given off is determined by weighing the cylinder.

Finally, the cylinder is completely emptied, and the nature and quantity of the residue determined.

The results of these analyses are given in the following table:—

No.	Origin of the CO ₂ .	Water per cent.	Gases insoluble in soda. Per cent by volume.	Nature of the gas.
1.	Ozouf's process	0.07	2.0	Air
2.	Natural	0.10	0.8	Air
3.	Magnesite	0.13	4.0	CO
4.	Prepared artificially* ..	0.03	3.4	CO
5.	Carbonaceous rock	0.17	5.7	Air

* No more precise data could be obtained.

In the last case the composition of the residual gas was determined by means of pyrogallate of potash, and it was found to consist of 85.1 per cent of nitrogen and 14.9 per cent of oxygen, by volume; it was thus richer in nitrogen than air is.

On repeating the estimation of the gaseous impurities, after having allowed a certain quantity of gas to escape, the following results were obtained:—

No.	Gas given off.	Percentage of gaseous impurities.
1.	Half	Inappreciable
3.	Do.	Do.
4.	90 per cent.	Do.
5.	33 ..	0.8 per cent by volume.

Tube No. 2 was not examined again, as the original proportion was so low.

The amounts of carbonic acid and residue were as follows:—

No.	Contents.	Residue.	Nature of the residue.
1.	10.0 kilos.	0.5 g.m.	Water, with traces of oxide of iron in suspension.
2.	10.3 ..	520.0 grms.	517 grms. of water and 3 grms. of oxide of iron.
3.	10.1 ..	1.0 gm.	Water, with traces of oxide of iron in suspension.
4.	8.0 ..	0.0 ..	—
5.	9.9 ..	8.5 grms.	Water, with traces of oxide of iron in suspension.

We see, from these analyses, that the purity of commercial carbonic acid is very satisfactory. The proportion of water in the gas which comes off in the first place is negligible. The gaseous impurities are of more importance: from this point of view the natural gas is the purest, but in the others the total amount is low, and should not exceed half the figures given, since, when the cylinders are half empty, we can no longer determine the amount present. The presence of carbonic oxide in the gas made from magnesite seems to indicate that it was not prepared only by the use of acid, but also by heating the mineral, and that the carbonic oxide is formed in the presence of reducing materials. The carbonic oxide is to be reckoned with when the carbonic acid is for use in ice-making machines on board men-of-war, these machines being liable to leak especially when first put to work, and the space in which they are used is very restricted. I have therefore advised that the carbonic acid must be free from carbonic oxide; it should not, in fact, contain any other impurity than air. The residue was not of any importance, except in the case of the natural gas (5 per cent by weight); in the other cases it does not exceed 0.1 per cent. I cannot give any other reason for the high proportion of water in the natural gas than that given by the maker, viz., that during the manufacture of mineral

water a certain quantity of water enters the cylinders, and care should be taken to empty this out each time they are used.—*Zeitschrift für Angewandte Chemie*, April 17, 1900.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JULY 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES FERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, August 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from July 1st to July 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us during the month, all were found to be clear, bright, and well filtered.

There has been very little rain at Oxford during the month, the total fall being only 0.88 inch, distributed over ten days. The average fall being 2.68 inches, we have a deficit of 1.80 inches for the month. This cancels the previous excess of 0.96 inch for the year and gives instead a deficit of 0.84 inch, or 6.1 per cent on the thirty years' average.

Our bacteriological examinations of 520 samples have given the results recorded in the following table; we have also examined 36 other samples, from special standpipes, &c., making a total of 556 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 25 samples) ..	299
New River, filtered (mean of 132 samples) ..	11
Thames, unfiltered (mean of 26 samples) ..	1338
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 274 samples) ..	25
Ditto ditto highest	250
Ditto ditto lowest	0
River Lea, unfiltered (mean of 25 samples) ..	450
River Lea, from the East London Company's clear-water wells (mean of 38 samples) ..	19

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

Contribution to the Study of Roumanian Petroleum.—L. Edeleanu and G. A. Filiti.—The authors divide their subject into two parts. In the first part they describe the chemical examination, showing the true nature of the raw petroleum by the determination of the constitution of the bodies of which they are formed. In the second part they show the physical and technical properties, which are of such importance in the industry of petroleum refining.—*Bull. Soc. Chim.*, xliii., No. 10.

NOTICES OF BOOKS.

The Oil-Chemist's Handbook. By ERASTUS HOPKINS. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1900. Pp. 72. 8vo.

THE author of this volume holds the position of chemist in charge of United States laboratories at Boston, and has had many years' practical experience as an oil-chemist. As stated in the introduction, the handbook is intended to contain not only the methods used in testing and analysing oils, but also the analytical data for identifying unknown oils and for detecting adulterants. The small number of pages in the book gives to those who have not examined it no idea of the immense amount of information it contains; besides the 72 numbered pages there are many folding tables in which practical data are closely packed. For instance, the "Table of the Chemical and Physical Constants of Oils and Fats" has six openings, each about two feet long, ruled in sixteen columns. The position of the oils in the various tables is determined by the numerical value of the constants; thus in the table of iodine absorption constants, sardine-oil stands first, while in the table of acetyl values castor-oil heads the list. This arrangement is intended to facilitate the identification of an unknown oil. References to journal-literature are freely given. The author uses the objectionable word "titer," and writes benzol for benzene. The book will be welcomed by all engaged in testing oils. There are illustrations and an index.

Air, Water, and Food, from a Sanitary Standpoint. By ELLEN H. RICHARDS and ALPHEUS G. WOODMAN. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1900. Pp. 226. 8vo.

MRS. RICHARDS, who is Instructor in Sanitary Chemistry at the Massachusetts Institute of Technology, Boston, is already known to readers of the *CHEMICAL NEWS* by her earlier works: "Plain Words about Food," "Home Sanitation," "Food Materials and their Adulterations," "The Chemistry of Cooking and Cleaning," and "The Cost of Living as Modified by Sanitary Science." These volumes show that the busy author has long devoted her energies to the study of the three essentials for healthful human life; it is gratifying to note that her experience has made her hopeful for the future, for she writes:—

"The day is not far distant when a city will be held as responsible for the purity of the air in its school-houses, the cleanliness of its water in its reservoirs, and the reliability of the food sold in its markets as it now is for the condition of its streets and bridges; nor will the years be many before educational institutions will be held as responsible for the condition of the bodies as of the minds of the pupils committed to their charge; when a Chair of Sanitary Science will be considered as important as a Chair of Greek or of Mathematics; when the competency of the food-purveyor will have as much weight with intelligent patrons as the scholarly reputation of any member of the faculty."

The book under review contains, besides discussions of the characteristics of pure air, safe water, and wholesome food, instructions for securing them under many conditions, for proving them, and for detecting the commoner sophistications of the latter. In the chapters on Air and Ventilation, the author describes the methods of determining carbon dioxide in the atmosphere devised by Pettenkofer, by Cohen and Appleyard, by Fitz, and by Wolpert. The chapters on Water are written from the standpoint both of the householder and of the chemist, and contains much practical information. These are followed by a chapter giving methods for determining free and albumenoid ammonia, the total organic nitrogen, the carbonaceous matter, the chlorine, the dissolved oxygen, the hardness, &c. The chapters on Food in Relation to

Human Life, and the Problem of Safe Food, show the familiarity with the subjects that might be expected of the author; but she treats the analysis of milk, butter, cereals, and fermented liquors rather cursorily.

In the Appendix are useful tables and a list of books relating to the topics treated, chiefly in English. There is an index. The book will be found valuable as an educational factor in laboratory training, and as a handbook in the workshop of the analytical chemist called on to make examinations of air, water, and food.

H. C. B.

Commercial Organic Analysis. By ALFRED H. ALLEN, F.I.C., F.C.S., &c. Third Edition, with Révisions and Additions by the Author and HENRY LEFFMANN, M.A., M.D. Volume II., Part II. London: J. and A. Churchill. 1900. Pp. 330.

THE present volume completes the revision of volume II. of this work, with the exception of the section relating to essential oils. It deals with the hydrocarbons, petroleum and coal-tar products, asphalt, phenols, and cresotes.

The original second volume, dealing with oils of all kinds, consisted of 573 pages. It has now been split up into three Parts. Part I. was published in 1899 (see CHEM. NEWS, vol. lxxix., p. 225), Part II. is now before us, and Part III., which will treat of the essential oils, aromatic acids, camphors, resins, and balsams, is now ready for the printer. The two parts already issued cover over 700 pages, so it will be seen that a large amount of matter has been added.

The principal additions and changes herein made are:—The systematic arrangement of the hydrocarbons according to the modern system, including the application of the nomenclature of the Geneva convention—this is a heavy task in itself; we also find summaries of the technology of acetylene, coke-oven tar, and asphalt; the testing of petroleum, lubricating oils, and coal-tar products, and special tests for many benzene derivatives now used as drugs, food-preservatives, or disinfectants. A large addition has also been made to the analytical data concerning phenol, phenolic disinfecting agents, and wood-tar cresotes.

The reviser has added, as an Appendix, a detailed description of new extracting and drying apparatus, and has calculated tables of comparison of Beaumé degrees and specific gravities lighter than water. The Index, which is always an important feature in a book of this class, has been much enlarged, and makes a worthy ending to an excellent work.

The Chemistry of Fire and Fire Prevention. By HERBERT INGLE, F.I.C., F.C.S., and HARRY INGLE, Ph.D. (Munich), B.Sc. (Victoria). London: E. and F. N. Spon, Ltd. New York: Spon and Chamberlain. 1900. Pp. 287.

THE present work, intended more especially for the use of Insurance Surveyors, Works' Managers, and those interested in fire risks and their diminution, is based upon two courses of lectures delivered by one of the authors; these lectures, considerably extended and re-arranged, are now published in this volume.

On looking through the book it will soon be seen that the title is to a certain extent misleading; the Chemistry of Fire occupies only a small portion, principally in Chapter I., while the part devoted to fire prevention and extinction (Chapter XV.) is not much larger. The intervening chapters deal with a variety of subjects, such as the preparation and purification of coal-gas; fuel; illuminants; oils; volatile solvents and coal-tar products; paint- and varnish-making; textile manufactures, &c. In all these chapters the possible dangers of fire are pointed out, but the general impression left is more that of a work on chemical technology.

A very convenient innovation worth noting is, that the

heading of every left-hand page gives the number of the chapter as well as the subject.

The Cyanide Process of Gold Extraction. A Text-book for the Use of Mining Students, Metallurgists, and Cyanide Operators. By JAMES PARK, F.R.G.S., &c. First English Edition, Revised and Enlarged from the Third Edition published in New Zealand. London: C. Griffin and Co., Ltd. 1900. Pp. 127.

OF late years the application of scientific investigations and methods to the treatment of ores has rendered metallurgy more and more dependent on chemical knowledge, and in no department is this more striking than in the cyanide process of gold extraction. Quite recently wet-crushing and cyanide treatment have largely superseded dry-crushing and direct cyaniding in New Zealand, and in every case the change has been attended with complete success. The results obtained with these comparatively complex ores are of very great interest, and have been embodied by the author in a separate appendix.

The fact that gold and silver are soluble in solutions of alkaline cyanides has long been known, but the discovery that the solution must be *dilute* is comparatively modern, and is one of the most important facts in the application of this process to successful gold extraction on a large scale.

No hard and fast rules can be followed in the working of the cyanide process, as modifications have to be made to meet the special requirements of the different classes of ores and tailings; but the essential features of the process are practically the same in all cyanide plants, and may be summarised as follows:—1. Filling the leaching vats. 2. Preliminary treatment with water or alkaline washes if necessary, to remove the mineral salts and acids, in the case of an acid ore, which would decompose the cyanide solutions. 3. Leaching with what is called the "strong" solution of cyanide: this, however, should only contain from 0.3 to 0.6 per cent of KCy. 4. First wash with cyanide from "strong" sump, containing 0.1 to 0.25 per cent of KCy. 5. Second, wash with cyanide from "weak" sump, containing from 0.02 to 0.1 per cent of KCy. 6 and 7. Same as last. 8. Washing with clean water. The cyanide solutions are then allowed to flow through the zinc extraction boxes, where the gold is deposited in the form of a black powder or mud; this is then smelted and cast into ingots. The above process is generally known as the McArthur-Forrest process, and is most successful in the treatment of ores in which the gold occurs in a very finely-divided state, and in which the quantity of base minerals or metallic salts, destructive to cyanide, is small.

Since the introduction of the cyanide process, the precipitation of the gold by metallic zinc has always been regarded as a weak point, and much time has been spent in the endeavour to find a substitute for it. Electrical precipitation naturally engaged a good deal of attention, but it was found that the precipitation of the gold from weak solutions was accompanied by the decomposition of the water. In the Siemens-Halske process this difficulty is overcome by causing a slow artificial circulation of the cyanide solution in the extractor: this method has been successfully introduced in a number of mines in the Witwatersrandt gold-fields, and was, before the war, already becoming a formidable rival to the McArthur-Forrest zinc-precipitation process.

Other cyanide processes which may be mentioned are the Hannay electro-cyanide method, by which the whole of the gold is obtained as an amalgam; the Park-Whitaker cyanide process, intended for the treatment of cupriferous ores and concentrates, which are first subjected to a chloridising roasting,—after this the soluble copper chlorides are removed by leaching with water; an alkaline wash is then applied, and the gold and silver extracted with a dilute solution of cyanide. In the Sulman-Teed process cy-

anogen bromide, or some other halogen compound of cyanogen, is used as the solvent in conjunction with zinc as a precipitant; the process is claimed to be well adapted for the treatment of pyritic concentrates and complex ores.

We can strongly recommend this book to all those engaged in gold and silver mining: there will no doubt be a very large extension of mining operations when the pacification of the Transvaal has been completed, and the demand for really skilled managers and metallurgists will be greater than it has ever been before. The author is a thoroughly practical man, and has handled the subject in a clear and practical manner, while at the same time the theoretical side of the question has not been neglected.

CORRESPONDENCE.

ACTION OF SULPHUR CHLORIDE ON OILS.

To the Editor of the Chemical News.

SIR.—In a work, "Le Caoutchouc et la Gutta Percha," by Seeligmann and others, I note, at page 279, the following extraordinary statements, due to Henriquez, on the authority of the CHEMICAL NEWS (1888, p. 113):—

"Warren pretend que les huiles siccatives donnent avec la chlorure de soufre des masses solides, insolubles dans le sulfure de carbone, tandis que les huiles non siccatives donnent des produits solubles dans ce véhicule. Stollmann, dans la dernière édition du Dictionnaire du Muspratt, écrit que ces résultats semblent mériter peu de créance, puisque l'on sait que l'huile d'olives, type des huiles non siccatives, se transforme, sous l'action de chlorure de soufre, en une masse analogue au caoutchouc, insoluble dans l'éther. Les faits signalés dans le brevet de Sommers, ainsi que nos expériences personnelles, contredisent formellement les assertions de Warren."

I cannot find that the article in CHEMICAL NEWS contains anything to justify the conclusion that olive oil in particular does not give a product different to many other oils which are regarded as non-drying, as olive oil, castor oil, almond oil, cotton oil, &c.

In fact, the conclusion I came to when the article was written, was that the division of oils into drying and non-drying oils was of technical use to a painter, and as a scientific distinction could not be recognised.

This work of Seeligmann should be of use to your readers who are interested in indiarubber and gutta-percha. It can be obtained at 30, Rue des Dragon, Paris.—I am, &c.,

THOMAS T. P. BRUCE WARREN.

Tamworth Villa,
Earlham Grove, Forest Gate.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

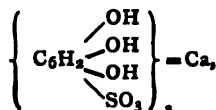
NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 8, August 20, 1900.

Extraction of the Oxygen of the Air by means of Solution at a Low Temperature.—Georges Claude.—The author's experiments had for their object the production of an air containing 50 or 60 per cent of oxygen, at a comparatively small cost, such a gas being sufficient for most purposes where the use of oxygen is necessary. The method employed consisted in saturating a quantity of certain liquids at a known pressure and temperature with oxygen, and then extracting the gas by lowering the

pressure. The author used several alcohols and ethers, besides acetone, acetol, chloroform, liquid methyl oxide, petrols, benzine, liquid chlorine, and different inorganic liquids. The results obtained are not entirely satisfactory, but the research is being continued.

Pyrogallol-Sulphonic Acids.—Marcel Delage.—By the action of sulphuric acid on pyrogallol, and purification of the products obtained, the author prepared a calcium salt of pyrogallol-sulphonic acid, in the form of large and small crystals. Both these sets of crystals dried at 110° correspond to the formula—



the experimental numbers closely approaching those of theory. The barium salt has also been obtained, and also the pyrogallol-monosulphonates of potassium, sodium, and ammonium.

The Dextrines of Saccharification.—P. Petit.—The author prepares the dextrines which are formed during saccharification of a starch sediment at 50°, 60°, and 70°, the action being stopped by a rapid heating, after which iodine ceases to produce a colouration.

The Employment of Sodium Dioxide to Purify Wells Contaminated by Carbonic Acid.—E. Derennes.—Several methods of removing the carbonic acid from wells have been tried, all more or less awkward. The use of sodium dioxide seems to solve the problem. The CO₂ is absorbed, and is replaced by an equal volume of oxygen, the air returning to its normal composition.

Nos. 9 and 10.

These two numbers contain no matter of chemical interest.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 10.

Action of Hydrogen on Sulphide of Antimony.—H. Pélabon.—Already noticed.

Selenide of Zinc and its Dimorphism.—M. Fonzes-Diacon.—Already noticed.

New Preparations of some Oxides of Uranium.—J. Aloy.—Already inserted in full.

On the Cyclic β-Diketones.—M. Georges Leser.—There is at present no general method for the preparation of these bodies, of which three only are yet known. The first two were prepared by Ad. von Baeyer (*Berichte*, xxx., 28) from the ketonic acids of menthone and tetrahydrocarvone; they are pentagonal. The author has prepared the third (*Bull. Soc. Chim.*, xxi., 546) by the isomerisation of acetylmethylheptenone. These three diketones have some common properties, but they have also differences which must not be overlooked. The first two form a salt of copper, and become hydrolysed quantitatively on contact with the alkalis, re-forming the original ketonic acids; the third only undergoes this hydrolysis partially, and a secondary reaction intervenes; it does not give a copper salt. In the endeavour to find a reason for these differences, the author has succeeded in reproducing a certain number of these bodies, and in applying Claisen's reaction to the cyclamones. Wallach's methylcyclohexanone being easy to prepare, and the author here describes the method applied to this ketone.

The Dichlorised Phthalic Acids, Products of Condensation.—E. C. Severin.—A long paper not suitable for abstraction.

Estimation of Chlorine in Gastric Juice.—G. Meillère.—The estimation of chlorine in gastric juice comprises the following operations:—(1) Preliminary filtration or centrifugation of the liquid for the subsequent

examination of the deposit. (2) Determination of the acidity on 10–20 c.c. by means of a titrated solution of baryta in the presence of phthalein. The result is calculated as HCl; it is convenient to also express it in cubic centimetres of normal solution. (3) The total chlorine: 10 c.c. of the juice, containing a slight excess of carbonate of lime, are treated with 20 to 50 centigrams. of nitrate of lime (according to the richness of the juice in organic matters), and evaporated to dryness, then gently ignited until the carbon has disappeared. The residue is taken up with a little distilled water containing a few drops of acetic acid; a little chalk and a drop of potassic chromate are added, and the liquor is then titrated with nitrate of silver. (4) Chlorine in the dry residue. This residue is best prepared in vacuo, and the operation proceeded with as in the case of the total chlorine. (5) Chlorine in the ash. Ten c.c. of the juice are evaporated and gently ignited without the addition of any other body, and the operation completed as above. The results are calculated as HCl.

Chemical Analysis of the Mineral Water from the Cold or Park Spring at Evaux-les-Bains (Creuse).—E. Bonjean.

Analysis of the Natural Mineral Water from the No. 3 Spring, Brault Sail-sous-Couzan (Loire).—E. Bonjean.—Both the above papers have been inserted in full.

New Method for the Estimation of Fats in Dairy Products.—M. Lindet.—This method of estimating the fatty matters in milk and cheese is based on the solubility of casein in a concentrated solution of resorcin. The apparatus designed for effecting this analysis consists of a cylindrical glass bulb, having a capacity of 15 grms. for milk analysis, and 18 to 20 grms. for cheese; it is closed at one end with an indiarubber stopper, through which a glass rod passes; the other end terminates with a narrow graduated tube, the special calibration and use of which is fully described. When in use this apparatus is placed vertically with the graduated tube downwards; this tube is plugged and filled with mercury to prevent the casein entering and making it dirty. Five grms. of resorcin, 5 c.c. of milk, 2 drops of soda at 36°, and 1 drop of solution of a colouring material are introduced; the stopper is then fixed in firmly, and the whole apparatus turned upside down to allow the mercury to fall out of the graduated end, which is then unplugged; it is then placed in a water-bath of sufficient depth for the graduated tube to be almost entirely immersed in the boiling water. The casein dissolves rapidly, especially if the apparatus is shaken, which must be done with care. It is left in the water-bath until the level of the butyrous layer does not alter after two readings made at an interval of ten minutes. The operation lasts half-an-hour. The results obtained are identical with those given by exhausting with ether. The estimation of the fatty matter in cheese is even more simple, and the separation of the two layers is done more easily. One grm. of cheese, about 15 c.c. of a warm solution of 100 grms. of resorcin in 100 c.c. of water are placed in the apparatus, which is then reversed and treated as before. The operation lasts from ten to fifteen minutes. If it is desired to estimate the fatty matters in cream, it is only necessary to dilute the cream with the quantity of water necessary to bring it to the original strength of the milk.

MISCELLANEOUS.

Schools of Chemistry.—The following additional information of interest to Students has been received:—

THE BIRKBECK INSTITUTION, Breams Buildings, Chancery Lane.—This Institution, which has now completed 77 years of educational work in the metropolis, commences its new Session on Monday, October 1st. There

are both day and evening courses of study, which comprise the various branches of Natural Science, Mathematics, classical and modern Languages, Economics, Commercial Subjects, Law, Mental Science, Music, and Art. The courses provide for the Examinations of the University of London, in the faculties of Arts, Science, and Commerce, those of the Conjoint Board, Civil Service, &c. The Report for the last Session shows that during the year fifty Students passed some university examination, while large numbers gained successes at various public examinations. The Institution has had many additions to its appliances in recent years, and the Physical, Chemical, and Metallurgical Laboratories are now very thoroughly equipped. The day classes provide courses in Chemistry, Biology, Physics, and Mathematics, for the Science Degrees of London University. There are also day classes in Languages, Music, and Short-hand. The School of Art is open both day and evening. During the recess, considerable additions and improvements have been made by the aid of a gift of 2000 guineas from Mr. F. Ravenscroft to commemorate his completion of a membership of fifty years. A new reading room, a new magazine room, and a social room have been provided, and a well-appointed Metallurgical laboratory has been added. The Calendar supplies complete syllabuses of the classes and full information about the Institution. A popular Lecture or Entertainment is given in the Theatre every Wednesday evening.

CHARTERHOUSE (WILLIAM ROGERS' MEMORIAL) SCIENCE SCHOOLS AND LITERARY INSTITUTE.—The Winter Session will commence Sept. 29th. During the late Session a large number of Students, mostly Elementary Teachers, availed themselves of the privileges afforded by this Institute. Instruction of a practical character is given in most of the Sciences at a nominal fee. Students who aim at becoming proficient in Chemistry (Organic and Inorganic) can work in a well-fitted Laboratory. Aspirants for University Honours can, at a small expense, be assisted in their studies. Special opportunities for the study of Hygiene will be afforded this Session to Laundry and Cookery Students who desire to be qualified for London School Board and L.C.C. appointments. Full particulars of the classes can be obtained from C. Smith.

CHEMICAL DEPARTMENT, MIDDLESEX HOSPITAL MEDICAL SCHOOL.—Lecturer, A. M. Kellas, B.Sc., Ph.D.; Demonstrator, W. G. Taylor. The Chemical Department opened Sept. 25th. Two complete courses of Lectures and practical work are given for the Conjoint Board Examination (R.C.S. and R.C.P.), starting in October and May respectively. A complete course of practical and theoretical work for the Inter M.B. of London University starts in January and continues to beginning of July. Two courses of Toxicology for the M.B. of London are given annually, commencing in September and July respectively. Courses of Chemistry for the Diploma of Public Health—Conjoint Board or Cambridge—can be started at any time throughout the Session, and hours can usually be arranged to suit the convenience of candidates. The Bacteriology in connection with this course is given by A. G. R. Foulerton, F.R.C.S., D.P.H.

THE MUNICIPAL TECHNICAL SCHOOL, Princess Street, Manchester.—The new School now in course of completion will be opened in September, 1901. In addition to the ample arrangements provided for Industrial Chemistry, the Committee have decided upon the erection of new building, 1248 square yards in area, for Bleaching, Dyeing, Printing, and Finishing Textile Goods. The new School, it is stated, will be the largest and probably the most completely equipped school in the kingdom.

HARTLEY COLLEGE, SOUTHAMPTON.—Principal, R. Wallace Stewart, D.Sc. (Lond.). London University Courses in Arts and Science, Science Courses for Medical and Dental Students, Mechanical and Electrical Engineering Courses.

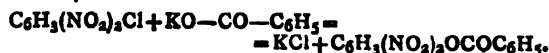
READING COLLEGE.—Lecturer in Chemistry, C. M. Luxmoore, D.Sc., F.I.C.; Assistant, J. L. E. Drugman, Ph.D.; Demonstrator, W. J. Monk. The Autumn Term begins Thursday, October 4th. The following are the Courses of Instruction in Chemistry:—I. Elementary Course (Autumn Term only), Tuesdays and Thursdays; fee £1 1s., or, including laboratory instruction, £2 12s. 6d. II. First Year Course, Tuesdays and Thursdays. III. Second Year Course, Wednesdays and Saturdays. IV. Advanced Course. V. Agricultural Chemistry, Mondays and Fridays. The Fees for the above, including Laboratory Instruction, are—Term, £4 4s.; Session, £8 8s. VI. Elementary Chemistry for Agricultural and Horticultural Certificate Students, Mondays; fee, £1 1s. VII. Pharmaceutical Chemistry. VIII. Pharmacy. IX. Laboratory work, daily; Fee, term, £3 3s. Special course of studies in Pharmacy. Special class for Teachers in Elementary Schools. Evening classes in Chemistry, Physics, and other Science subjects. The Diploma of Associate of Reading College in Letters and Science is awarded to Students who attend a full course of instruction during two Sessions, and pass specified Examinations. Full particulars of subjects of study and other information will be found in the College Calendar, to be obtained, post free 1s., from the Registrar.

INSTITUTE OF PUBLIC HEALTH AND TECHNOLOGY, Seymour House, Old Trafford, Manchester.—Principals, Charles Estcourt, F.I.C., F.C.S., &c., and Philip Anderson Estcourt, Ph.D. The course of instruction is especially intended for those who require a knowledge of Chemistry and Bacteriology bearing upon Sanitary matters. Chemical Technology is specially taught with a view to enable Medical Men and others engaged in Sanitary matters to understand the operations at the various works which may come under their control. Persons who desire to take up the branch of Chemical Engineering, including Electrical Engineering, will also have special opportunities of pursuing such studies.

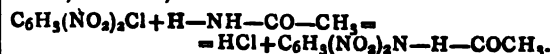
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Action of Chlordinitrobenzene on Benzoate of Potassium and on Acetamide.—O. Kym.—Alcoholic solutions of chlordinitrobenzene and of benzoate of potassium do not react on one another, either in the cold or when heated. If, however, we mix chlordinitrobenzene with benzoate of potassium in the powdered state, and heat the mixture to 180° on an oil-bath, a reaction takes place, and the dinitrophenylic ether of benzoic acid is formed,—



The ether in question melts at 132°; it is identical with that obtained by heating together dinitrophenol and chloride of benzoyl. Acetamide and chlordinitrobenzene also do not react on each other in alcoholic solution, either in the cold or when heated. When heated in the solid state for ten hours in an oil-bath at 200–210°, they give a large quantity of dinitraniline, with a return of 93 per cent of the amount of chlordinitrobenzene used. Acetyldinitraniline, which is saponified during the reaction, is formed,—



The author added acetate of sodium to the fused product, with the object of preventing the saponification of the acetyldinitraniline, but in this case he obtained dinitrophenol. This reaction may be explained by the supposition that dinitraniline is also formed, but that this latter has been transformed into dinitrophenol by the decomposed acetate of sodium, which acts as the carbonate. Chlordinitrobenzene heated with acetate of sodium alone gives rise to the formation of traces of dinitrophenol only.—*Berichte*, xxxii., 3539.

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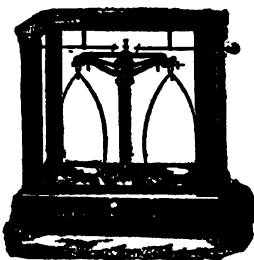
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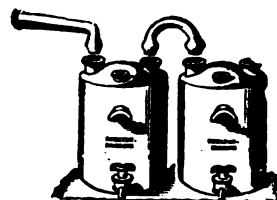
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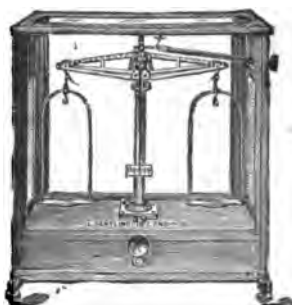
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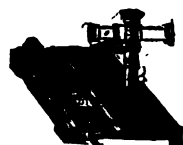
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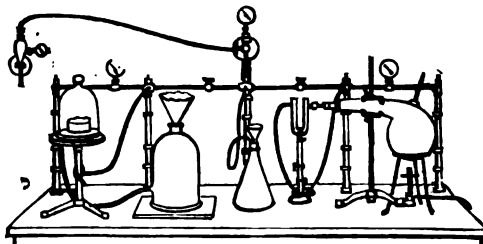
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SOME RECENT WORK ON THE DIFFUSION OF GASES AND LIQUIDS.*

By HORACE T. BROWN, LL.D., F.R.S.

THE author's recent work, in conjunction with Mr. F. Escombe, on the fixation of carbon by green plants from the carbon dioxide of the atmosphere, necessitated a re-examination of the process of *static diffusion* from a somewhat new standpoint, and in the course of the experiments some new and interesting facts were brought to light which have an important bearing on the laws of diffusion of gases and liquids.

In the case of carbon dioxide and air the absolute rate of the inter-penetration of one gas by the other has hitherto only been determined for high ratios of mixture, but it would be manifestly unsafe to apply the values of such coefficients to the static diffusion of atmospheric carbon dioxide, which only exists in the air to the extent of 0.03 per cent by volume. An attempt was consequently made to determine the coefficient of diffusivity of atmospheric carbon dioxide by a process of static diffusion.

If we imagine a vertical cylinder to be filled with atmospheric air containing the normal amount of carbon dioxide, and regard any plane surface within the enclosed air, it follows, from the kinetic theory, that in any given time the same average number of carbon dioxide molecules will cross this plane in opposite directions, and there will consequently be no change in the average distribution of the molecules throughout the cylinder. If, however, we imagine the bottom of the cylinder to become suddenly capable of absorbing carbon dioxide, say by the introduction of a layer of a solution of caustic alkali, then the molecules of this gas which strike the bottom of the cylinder will be wholly or partially trapped, and in the first brief interval of time a very thin stratum of air, parallel to and immediately above the absorbing surface, will be partially depleted of its carbon dioxide. If we now further consider the interchange of carbon dioxide molecules taking place between this partially depleted layer and the one above it, it is evident that now in a given interval of time a larger number of the molecules must pass from the higher to the lower stratum than from the lower to the higher; the "balance of exchange" will consequently be in favour of the lower layer. This is a condition which will be rapidly propagated from below upwards, and, providing the air outside the cylinder is maintained of uniform composition, a *static condition* will be established in the column. When this condition has been reached, the uncompensated balance of exchange between any two contiguous layers will constitute a steady *flux* or *drift* of carbon dioxide from the outer air to the absorbent surface, the rate of which, if the surface is perfectly absorbent, will be conditioned solely by the diffusivity of the gas. The question from the mathematical standpoint can now be treated in exactly the same manner as the "flow" of heat in a bar after the permanent state has been reached, or the "flow" of electricity between any two regions of a conductor maintained at a constant difference of potential.

The diffusivity of atmospheric carbon dioxide has been determined on these lines by the author and Mr. Escombe, and has been found to have a mean value in C.G.S. units of 0.157. It is pointed out, however, that the difficulties of eliminating all disturbing causes are very great, but

that the experiments suffice to show that the inter-diffusivity of carbon dioxide and air in the ratio of mixture in which they occur in the atmosphere does not materially depart from the value obtained by Loschmidt and others with much higher ratios of mixture. Another and more accurate method subsequently described confirms this conclusion.

An account is then given of the author's researches on the diffusion of gases and liquids through apertures in a diaphragm.

If a condition of static flow of atmospheric carbon dioxide is established down a short column of air, towards a perfectly absorbent surface, the rapidity of flow, conditioned by the diffusivity of the gas, is strictly proportional to the area of the cross section of the column. If, however, we partially obstruct the open end of the column with a diaphragm pierced with a single central aperture, or if we introduce such a diaphragm anywhere in the line of flow, diffusion is modified in a remarkable and unexpected manner.

As the aperture in the septum is gradually reduced in size, the flow through unit area is accelerated, and when the rim around the hole has a breadth of two or three times the diameter of the aperture, this acceleration is found to vary inversely as the diameters of the opening. In other words, the amount of flow then becomes directly proportional to the *linear dimensions* of the opening.

The same fact was also shown to hold good for the diffusivity of aqueous vapour in air, either by the static diffusion of the vapour down a column of air, or by the evaporation from dishes which were covered by thin plates of mica perforated with holes of a definite size. The "diameter law" was also proved for diffusible salts in solution in a somewhat similar manner, the mobility of the liquid being destroyed by using a small percentage of gelatine.

When a solution of caustic alkali is exposed to air which is in slight motion, the amount of carbon dioxide absorbed in a given time is proportional to the *areas* of the surface exposed; but if the liquid surface is surrounded with a broad rim in the same plane, and precautions are taken to prevent air-currents, it is then found that absorption takes place proportionately more rapidly with the small discs of liquid. When extreme precautions are taken, and comparatively small surfaces are employed, a very near approach to the "diameter law" can be attained, but it is more difficult to obtain this result with plane absorbent surfaces than it is with apertures in a septum, for reasons which will be seen later.

The explanation of the phenomena so far described is as follows:—

If we consider the simple case of a circular disc of liquid, surrounded by a broad rim and capable of absorbing carbon dioxide, when such a disc is exposed to perfectly still air, the convergent lines of carbon dioxide will creep through the air towards the disc, establishing a steady gradient of density, and this creep will be a flux perpendicular to the lines of equal density, which will thus form curved surfaces or "shells" surrounding the disc and terminating in the rim. The case is analogous to that of an *electric field* in the neighbourhood of a conductor, having the same shape and dimensions as the absorbent disc. The curves or "shells" of the gas of equal density are the analogues of the similarly curved lines of *equipotential* above the surface of the electrified disc, while the converging lines of creep or flux of the gas are the analogues of the *lines* or *tubes of force*. The partial pressure of the carbon dioxide in the shells of equal density must vary from *zero* at the absorbent surface to a density corresponding to the maximum pressure of the carbon dioxide in the atmosphere, a point which is theoretically at an infinite distance, but which is practically reached at a distance of five or six times the diameter of the disc. From these considerations it follows that the gradient of density, on which the diffusive flow depends,

* Abstract of a Paper read before the British Association (Section B), Bradford Meeting, 1900.

must vary inversely as the diameters of the discs, and that the absorbing power of the discs will vary directly with their linear dimensions, a deduction which is completely verified by experiment.

In the case of a diffusive flow through a circular aperture in a diaphragm, the hyperbolic lines of flow, which are *convergent* as they approach the aperture, bend round their foci situated in its edges, and form a *divergent* system on the other side. On the inner side of the diaphragm there is produced a system of density shells similar to those outside, but with the gradient of density in the contrary direction. This system of shells is termed *negative*, and is as effective as the outer *positive* system in regulating the flow according to the diameter law, so that this law still holds good even if the outer air-currents are sufficient to sweep away the external positive shells completely. This fact explains why it is easier to obtain a demonstration of the "diameter law" by diffusion through apertures than by diffusion into an absorptive disc formed by a solution of caustic alkali.

By diffusing colouring matter through apertures in a diaphragm the "density shells" can be rendered visible, and it may be shown that their ellipsoidal form is exactly that which is demanded by the above hypothesis.

Simple mathematical expressions were given for all the possible cases of diffusion through apertures in thin plates, and in plates of a sufficient thickness to constitute tubes.

Diffusion through multi-perforate diaphragms was next considered, the septa employed being thin celluloid plates pierced with small circular holes of a determinate diameter.

Such septa produce a local disturbance and intensification of the gradient of density in their immediate neighbourhood, so that the velocity of the flux increases as the obstruction is reached, and decreases again when this is passed. The result of this is that it is found possible to perforate a thin plate with a series of very small holes in such a manner as to exercise but little obstruction when it is interposed in a line of diffusive flow, although the aggregate area of the small holes may represent only a small fraction of the total area of the septum. Examples were given, and also formulæ for the calculation of the amount of resistance produced by a septum of a given thickness and perforated with holes of a definite size and distribution. The diffusive flow observed experimentally under these conditions is in close accord with that demanded by theory.

This method of diffusion through apertures affords a new and apparently accurate method of determining *coefficients of diffusivity*. The author and Mr. Escombe have, up to the present time, applied it to the cases of atmospheric carbon dioxide, aqueous vapour, and solutions of sodium chloride.

ON THE DISTRIBUTION OF CHLORINE IN WEST YORKSHIRE.*

By WILLIAM ACKROYD, F.I.C., Public Analyst for Halifax.

THE present observations are to be regarded as the preliminaries to an attempt to construct isochlores for this part of Yorkshire. The subject is one of acknowledged importance. Prof. Mason remarking:—"State maps, such as those issued for the States of Massachusetts and Connecticut, are most valuable, and their construction is well worth the expenditure of public money" (W. P. Mason, "Examination of Water," p. 29). Although the work is not far enough advanced for map construction, the figures which follow and observations thereon will be

of chemical and hygienic interest during the visit of the British Association to Bradford.

In the first place, these British normal chlorine figures are very high in comparison with the published American data. The lowest Massachusetts figure is 0.07 part of Cl per 100,000 in the area farthest removed from the Atlantic sea-border. Here the lowest found has been 0.7 part per 100,000 in the upper reaches of the Wharf, and all round it appears that the Yorkshire figures are about ten times larger than those of the State of Massachusetts, which is to be accounted for (1) by the closeness of the sea-border on either side to the Pennine range, and (2) by the density of population in, and the antiquity of, the inhabited areas.

The unit isochlor area is co-extensive with the highest hills and their becks, deans, and gills. The following chlorine determinations with waters from the upper reaches of the Wharf, Wenning, Ribble, Aire, and Calder will be sufficiently illustrative:—

	Chlorine. Parts per 100,000.
Barden	1.0
Grimwith Beck	1.2
Gate-up-Gill	0.7
Bleaback	1.0
Buckden Pike	1.0
Starbottom	0.8
Kettlewell	1.0
Buckden Village	1.2
Gaping Ghyll	1.2
Beck Head, Clapdale	1.3
Ingleborough Cave	1.2
Lower Benthams	1.4
Malham Tarn Outlet	1.0
Malham Cove	0.95
Airehead	1.0
Old Smelt Mill	1.1
Gordale Beck	1.2
Hanlith Bridge	1.1
Hardcastle Craggs	1.1
Walshaw Dean	1.1

As the sea is approached, and more populous districts are reached, the chlorine rises, and remarkable examples of rise may be met with on the same hill slope. Halifax furnishes a striking instance. The town rests on a sloping bed of millstone grit, the ancient and most thickly populated part being towards the bottom of the incline. The ground waters of the highest parts (Mount Tabor) vary from 1.6 to 2.6; widely separated wells about half-way down yield the figure 3.8; while, towards the bottom of the slope, two wells give the figures 4.7 and 5.5. The public water supply from Pennine gathering grounds, 10 miles away, stands at 1.3.

The figures obtained for other parts of the West Riding have not yet been collated, and are therefore reserved for a further communication.

ON A LIMITING STANDARD OF ACIDITY FOR MOORLAND WATERS.*

By WILLIAM ACKROYD, F.I.C., Public Analyst for Halifax.

MANY large towns, more especially in the West Riding of Yorkshire, have their public water supplies of Moorland origin. The case of Milnes v. the Huddersfield Corporation in 1881 gave great prominence to the fact that this class of water may give rise to plumbism. No satisfactory explanation could be given at the time, and it is only during the present decade that it has been clearly understood that the plumbo-solvent action of moorland waters

* Read before the British Association (Section B), Bradford Meeting, 1900.

* Read before the British Association (Section B), Bradford Meeting, 1900.

is to be associated with acidity. An idea of the relation is furnished by the following determinations:—

Parts per 100,000.			Lead dissolved from 4-inch piping.	
Acidity in equivalent of sulphuric acid.		In 1 hour	..	..
1.	0.29	..	0.03	..
2.	0.30	.. 1 ..	0.057	..
3.	0.29	.. 1 ..	0.025	..
4.	1.34	.. 1 ..	0.71	..
5.	1.59	.. 1 ..	0.95	..

The acidity is due to carbonic anhydride and peaty acid, and the total is found by ascertaining the number of c.c. of N/100 alkali required to neutralise 100 c.c. of the water, the result being expressed as sulphuric acid. Phenolphthalein is used as the indicator.

The acidity may be very high, as from peaty gathering grounds of small incline, say 1 in 44; or comparatively low in gathering grounds of steep incline, say 1 in 12 (Ackroyd, *Journ. Chem. Soc.*, 1899, p. 199).

In the former case, violent action on lead precludes its use for domestic consumption, and in the latter even a limit must be placed on the degree of acidity allowable. During epidemics of plumbism in the West Riding, much diversity of opinion has been expressed on various points connected with the matter which the author cannot discuss here; he contents himself with stating that, after some years of experience, he has never learnt of the occurrence of any case of plumbism where the acidity of the water has been under the equivalent of 0.5 part of sulphuric acid per 100,000 of water, and this he tentatively proposes as a limiting standard of acidity for potable waters of moorland origin when the acidity is determined in the manner already described with phenolphthalein as indicator.

The average acidity of nine samples of water not above suspicion in this respect was 0.63, ranging from 0.53 to 0.91; while, on the other hand, sixty-one samples above reproach, from neighbourhoods where plumbism has not been known, had an average acidity of 0.27, the extremes being 0.20 and 0.41.

INTERACTION OF FURFURALDEHYDE AND CARO'S REAGENT.*

By C. F. CROSS, E. J. BEVAN, and J. F. BRIGGS.

In a previous paper (*Chem. Soc. Journ.*, 1899, p. 747) it was shown that hydrogen peroxide reacted with furfural to form a monohydroxy-furfuraldehyde as the main product, with simultaneous production of the corresponding carboxylic acid in small quantity. The substituting groups appear to occupy the $\alpha\beta$ positions in the ring.

The monohydroxy-furfural is identified by means of its very characteristic reactions with phloroglucinol and resorcinol, as a constituent of the lignocelluloses.

Caro's reagent, prepared from potassium persulphate and sulphuric acid according to the directions of Baeyer and Villiger (*Ber.*, 1899), reacts under similar conditions in a different manner. In the first place the reaction appears to be quantitative, and when the furfural has taken up O_2 , a trace of either reagent, in excess, persists. The temperature remaining at 15–20°, and there being no evolution of gas, we may expect to find a product of empirical formula, $C_5H_4O_6$, and as the aldehydic group disappears this should be a hydroxypyromucic acid. On reduction with sodium amalgam an aldehydic product is obtained with the brilliant colour reactions of the monohydroxyfurfurals. Control observations on pyromucic acid proved that this acid is reduced under similar conditions to furfural.

The reactions of this hydroxyfurfural, though similar to those described in our previous paper, are sufficiently differentiated to indicate that we have obtained a second isomeride. Moreover, the corresponding acids are differentiated—the one giving Pb and Ba salts insoluble in acetic acid; the salts of the new acid are soluble, and, moreover, the acid undergoes hydrolysis with such ease as to make its isolation in the pure state a matter of great difficulty. In the course of the usual processes for isolating the Ba and Ca salts, decomposition occurs, and the crystalline salts isolated are those of a dibasic acid. On boiling the original product in solution at constant volume, formic acid distils continuously and with traces only of other acids. The yield of formic acid amounted in one experiment to 0.7 grm. per 1 grm. of original furfural. All these observations indicate that the original product of oxidation is the acid $C_5H_4O_6(COOH)(OH)_{\alpha\beta}$. A characteristic reaction of this original acid is the production of a yellowish-red precipitate with ferric chloride, similar to that obtained with pyromucic acid.

It is to be noted that the Caro reagent oxidises the constituent of the lignocelluloses which gives the brilliant colour reactions with phenols characteristic of these natural products, which we know to be a hydroxyfurfural. In this respect the reagent differs from the oxidants ordinarily used for bleaching purposes, e.g., hypochlorites and permanganates.

A quantitative experiment with a typical aldose (dextrose) and the Caro reagent, gave the somewhat unexpected entirely negative result. The cupric reduction (Fehling solution) was unaffected.

The typical ketose levulose, on the other hand, is slowly oxidised.

All of these matters are under investigation.

ON A SIMPLE AND ACCURATE METHOD FOR ESTIMATING THE DISSOLVED OXYGEN IN FRESH WATER, SEA-WATER, SEWAGE EFFLUENTS, &c.*

By Professor LETTS, D.Sc., Ph.D., &c., and R. F. BLAKE, F.I.C., F.C.S., Queen's College, Belfast.

AFTER criticising the existing methods for determining the dissolved oxygen in water volumetrically, the authors describe a very simple and accurate method for the purpose, of which the following is an outline:—

An ordinary separating funnel is filled with the water to be examined, and a measured volume withdrawn. A definite volume of standard ferrous sulphate solution is then added, and afterwards ammonia—the volume of these two reagents together corresponding with that of the water removed—and the stopper of the funnel is then inserted, care being taken that no air bubbles are enclosed.

Within the separating funnel there is now a layer of ferrous sulphate below, next the water, and above all the ammonia. These are mixed by inverting the vessel once or twice by a swinging motion, when a greenish turbid mixture results, which rapidly darkens as the dissolved oxygen is absorbed. After fifteen minutes the vessel (still stoppered) is inverted, and its tube or lower extremity (now, however, the upper one) is nearly filled with a mixture of equal volumes of sulphuric acid and water. The tap is then opened, when the acid flows downwards into the alkaline mixture, and in the course of a few minutes dissolves the iron hydrates, forming a clear solution. This is then run off into a porcelain dish, and there titrated, either with permanganate or bichromate, conveniently of the strength 1 c.c. = 1 c.c. of dissolved oxygen at N.T.P.

In the authors' experiment the separating funnel had a capacity of 332.5 c.c., practically $\frac{1}{3}$ litre; and its tube

* Read before the British Association (Section B), Bradford Meeting, 1900.

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Liquid analysed.	Titrations made with—	Dissolved oxygen per litre of fluid.		Difference.
		Found.	True amount.	
Distilled water saturated with air at 13.7°	Potmanganate	7.26	7.20 (Roscoe and Lunt) ..	+0.06
Belfast water (town supply)	Potmanganate	5.80	5.75 (Gasometric analysis)	+0.05
Sea-water from Belfast Lough	Potmanganate	5.53	5.10 (Gasometric analysis)	+0.43
Sea-water from Belfast Lough	Bichromate ..	5.10		0.00
Sewage effluent from "bacteria beds" ..	Potmanganate	0.66	0.36 (Gasometric analysis)	+0.30
Sewage effluent from "bacteria beds" ..	Bichromate ..	0.42		+0.06

contained, when nearly full, about 8 c.c. of diluted sulphuric acid. About 7 c.c. of the water was removed, 5 c.c. of standard ferrous sulphate solution added, and about 2 c.c. of strong ammonia. The ferrous sulphate solution contained about 12 grms. of the crystallised salt in 250 c.c. of distilled water. It was standardised for each determination by titrating 5 c.c. in a porcelain basin, mixed with the same volume of the water under examination as employed in the dissolved oxygen determination and the same volume of acid.

For all practical purposes the dissolved oxygen contained in the volume of water which the separating funnel holds amounts to the difference between the burette readings for the blank experiment and for the actual determination. The accompanying table gives a few typical results.

It will be seen that for sea-water and sewage effluents bichromate gives more accurate results than permanganate.

THE SEA-WEED *ULVA LATISSIMA* AND ITS RELATION TO THE POLLUTION OF SEA-WATER BY SEWAGE.*

By Professor LETTS, D.Sc., Ph.D., and JOHN HAWTHORN,
B.A., Queen's College, Belfast.

For a number of years past a very serious nuisance has arisen from the sloblands of the upper reaches of Belfast Lough during the summer months, the stench at low tide being quite overpowering, and the air heavily charged with sulphuretted hydrogen. A precisely similar nuisance, though not of the same magnitude, also arises from the sloblands in the northern portion of Dublin Bay.

The nuisance is caused by deposits of the green sea-weed, called *Ulva latissima*, or sea-lettuce, which in the two localities mentioned grows in abundance, and during high winds or gales is washed ashore. In Belfast Lough the quantity thus deposited is enormous, forming banks which are often several feet thick, and extend for miles along the coast. Once deposited, these layers of sea-weed often remain more or less stationary for months in the shallow bays or pools of the neighbourhood, and in warm weather rapid putrefaction occurs, and a perfectly intolerable stench arises, which is perceptible over a wide area, and seriously affects not only the comfort of the inhabitants of the district, but the value of their property also.

The Chemical Changes which occur when *Ulva latissima* Ferments in Sea-water.—An extended investigation in the laboratory has shown that when the sea-weed ferments, hydrogen and carbonic anhydride are first evolved in about equal proportions by volume, and fatty acids are produced. Isolation and examination of these latter show that propionic acid is the chief; butyric acid is also formed, and probably acetic acid in addition. In a later stage of the fermentation sulphides are formed, probably by reduction of sulphates present in the sea-water, and the sea-weed blackens from formation of ferrous sulphide, and this latter disengages sulphuretted hydrogen by the action of the fatty acids, whence the nuisance.

Composition of *Ulva latissima*.—The mean results of the analysis of the dried sea-weed were as follows:—

Carbon	35.15	{ Sulphur 3.21 Iron .. 2.20
Hydrogen	5.27	
Nitrogen	6.25	
Oxygen	37.96	
Ash	15.37 containing	
	100.00	

Attempts to isolate any definite principles by the methods of proximate analysis were not very successful, and no carbohydrate beyond cellulose could be identified.

Bacteriological Examination.—Considerable difficulty was experienced in attempts to isolate the organisms concerned in the putrefactive processes, and the ordinary methods failed for the greater part. The evidence at present collected points to there being two chief species concerned. (1) A spore-forming bacillus which apparently infects the sea-weed and causes the production of fatty acids from its tissues; and (2) a second micro-organism, probably derived from the mud of the shore, which gives rise to the formation of sulphides by a later fermentative process.

***Ulva latissima* in Relation to Sewage Pollution.**—The evidence tending to prove that the occurrence of the sea-weed in quantity in any locality is associated with sewage pollution is of three kinds:—

(1). **The Composition of its Tissues.**—The amount of nitrogen it contains is far in excess of that of any other sea-weed of which analyses are recorded. It contains over 6 per cent, and resembles in nitrogen content an animal product rather than a vegetable. (Dry cheese contains about 7, and dry milk about 5 per cent, whereas peas only contain about 4 and meadow hay 2 per cent).

(2). **Assimilation Experiments.**—A frond of the sea-weed was placed in various mixtures in succession, (a) of polluted sea-water, (b) polluted sea-water plus sewage, (c) polluted sea-water with sewage and ammonium salt, (d) sea-water plus nitrates. By determinations of free and albumenoid ammonia, and nitrates in these mixtures before and after the sea-weed had been in contact with them, the power of assimilating nitrogen which the weed possesses was ascertained and was found to be remarkably high. Thus in one experiment it absorbed the whole of the free ammonia from a polluted sea-water containing 0.050 part per 100,000 in seventeen hours. Nitrates were also rapidly absorbed, but not albumenoid matters. The weed remained in perfect health during these experiments and is still growing, although nine months have elapsed since the experiments were commenced.

(3). **The Localities where *Ulva latissima* abounds, and from which it is virtually absent.**—As stated above, the sea-weed is present in abundance in Belfast Lough, but it is almost entirely absent from Strangford Lough, which is similar in area and in many other respects to Belfast Lough, but differs from it in not being extensively polluted by sewage. In Dublin Bay the sea-weed is found in quantity in the harbour, which is highly polluted, but not in the southern parts of the bay, which receives no sewage, except near Kingstown, where a large sewage tank discharges on the ebb tide. There the weed occurs.

The evidence which the authors have collected tends therefore to the conclusion that the occurrence of *Ulva*

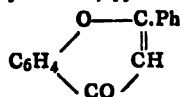
* Read before the British Association (Section B), Bradford Meeting, 1900.

latissima in quantity in a given locality is an indication of sewage contamination, and there can be no doubt as to the power which the weed possesses of absorbing nitrogen compounds from polluted sea-water. While thus acting as scavenger it may itself give rise to a very extensive nuisance.

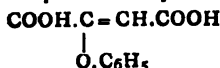
ON THE SYNTHESIS OF BENZO- γ -PYRONE.*

By Dr. S. RUHEMANN and H. E. STAPLETON, B.A. (Oxon).

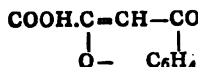
THE important group of yellow vegetable dyes, the chief of which are chrysin, fisetin, and morin, are derived primarily from a phenyl benzo- γ -pyrone—



This was synthesised in 1898 by Kostanecki, but the mother substance itself, benzo- γ -pyrone, had not, up to the middle of 1900, been isolated. The authors succeeded in preparing this compound from phenoxy-fumaric acid—



This acid dissolves with evolution of heat in concentrated sulphuric acid, and on diluting the solution with water a new acid is precipitated which was found to be benzo- γ -pyrone carboxylic acid—



On distillation in vacuo this broke up with the evolution of carbon dioxide, and a liquid distilled over which slowly solidified. It crystallised from a mixture of benzene and ligroin in flat needles which melted at 59° and analysis showed it to be benzo- γ -pyrone. The yellowish solution of benzo- γ -pyrone in concentrated sulphuric acid possesses a violet fluorescence.

A SIMPLE METHOD FOR COMPARING THE "AFFINITIES" OF CERTAIN ACIDS.*

By HENRY J. HORSTMAN FENTON, F.R.S.,
and
HUMPHREY OWEN JONES, B.A., B.Sc.

In a former communication (*Trans. Chem. Soc.*, lxxvii., 1900), the authors have described the isolation and properties of free oxalacetic acid, and several interesting reactions of this acid are now being investigated. During a more extended study of the *hydrasone* the following somewhat remarkable behaviour has been observed. Heated with dilute sulphuric acid it is transformed, as was previously shown (*Trans. Chem. Soc.*, lxxvii., 1900), into Wislicenus's phenylpyrazolon carboxylic acid—



but in order that this change may be complete it is now found that a certain minimum concentration of the acid is necessary. When heated with pure water an entirely different result is obtained: carbon dioxide is evolved, and the *hydrasone* of pyruvic acid separates in the crystalline state— $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_4 = \text{C}_9\text{H}_{10}\text{N}_2\text{O}_2 + \text{CO}_2$. If the concentration of the acid falls below this necessary minimum both reactions occur simultaneously, even though the acid is present in excess; with decinormal sulphuric acid, for example, about 26 per cent undergoes the second change.

* Read before the British Association (Section B), Bradford Meeting, 1900

A preliminary set of observations has been made with the following acids, using decinormal solutions and measuring the evolved carbon dioxide in a specially constructed apparatus—*hydrochloric, nitric, sulphuric, trichloroacetic, tartaric, malic, succinic, citric, acetic*. The results obtained indicate that the amounts of carbon dioxide evolved are in the inverse order of the concentration of the hydrogen ions, so that a comparison can be made of the relative "strengths" or "affinities" of the acids. The order obtained with the above acids agrees remarkably well with that resulting from the other well-established methods.

DERIVATIVES OF METHYL-FURFURAL.*

By HENRY J. HORSTMAN FENTON, F.R.S.,
and
Miss MILDRED GOSTLING, B.Sc.

THE authors have previously shown that an intense purple colour results when *laevulose, cane sugar, sorbose*, or *inulin* is acted upon by dry hydrogen bromide in ethereal solution. The colour-giving substance was isolated in a crystalline state and was shown to be *bromo-methyl-furfural*, its formation being characteristic of ketohexoses, or substances which yield them on hydrolysis. This substance is now the subject of further investigation, and the following results have so far been obtained:—

When acted upon by stannous chloride in acid solution the bromine is easily replaced by hydrogen, and the resulting liquid is identical in every way with δ -methyl-furfural; so that the reaction affords by far the simplest method for the preparation of the latter substance in a pure state.

The bromo-compound, when dissolved in appropriate solvents, readily reacts with various silver salts, giving rise often to beautifully crystalline compounds; the acetoxy- and benzoxy-derivatives have, for example, been prepared and analysed. By the action of sulphurous acid, these, like the parent compound, yield the remarkable condensation product $\text{C}_{11}\text{H}_{10}\text{O}_4$, which gives beautiful colour-reactions with caustic alkalis and with aniline. This condensation-product has also been further studied, and the results so far favour the author's original suggestion that it is a dicarbonyl compound.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING AUGUST 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES FERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolitan Water Act, 1871.

London, September 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from August 1st to August 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

* Read before the British Association (Section B), Bradford Meeting, 1900.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month has been considerably more than it was in July; 3.13 inches have fallen, the average being 2.32 inches; we have therefore to record an excess of 0.81 inch of rain for the month. This reduces the previous deficit for the year to 0.03 inch, or 0.18 per cent on the thirty years' average.

Our bacteriological examinations of 544 samples have given the results recorded in the following table; we have also examined 39 other samples, from special standpipes, &c., making a total of 583 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	94
New River, filtered (mean of 130 samples) ..	4
Thames, unfiltered (mean of 26 samples) ..	2349
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 297 samples) ..	9
Ditto ditto highest	106
Ditto ditto lowest	0
River Lea, unfiltered (mean of 26 samples) ..	138
River Lea, from the East London Company's clear-water wells (mean of 39 samples) ..	8

Of the 466 samples of filtered water supplied to London, examined bacteriologically during the last month, only one sample contained over 100 microbes per c.c. This shows that the usual efficient filtration has been more than maintained.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

ON THE TRANSMUTATION OF PHOSPHORUS INTO ARSENIC AND ANTIMONY.

By F. FITTICA.

I MUST beg to be allowed to make a few remarks on the communication made by M. Winkler (see CHEM. NEWS, lxxxi., p. 304), who contests the possibility of the transmutation of phosphorus into arsenic. It is more or less true that the experiments I made at the Chemical Institute at Marbourg have a slightly alchemistic appearance, but unfortunately I must confess that, even at the present day, I maintain the opinion I defended many years ago. At the time to which I refer, I said, at a conference at which I was present, "At heart we are still alchemists, not actually in the sense of making gold, but from the point of view of the possibility of transmuting metals. If it is true, and we have a right to suppose it so, that the metals as we know them are not elements, if it is true that there is only one element, as suggested by v. Helmont, or that there are even four, as imagined by the ancients, we ought to succeed, in the more or less near future, in transmuting the elements, and thus solving the old problem of alchemy." I made the above statement about twelve years ago. I also added that we had no need of the philosopher's stone for effecting the transmutation of metals. Since that time I have become more and more persuaded that the simplest thing is the most perfect, that nature has recourse to the most simple means, that the force of affinity combining atom to atom and molecule to molecule only requires certain elements

for its manifestation. What fault can be found in that? Is it not, on the contrary, the duty of the chemist and the naturalist to study the harmony of the universe, to show that the marvels of nature are accomplished by the aid of means, forces, and materials of the most simple kind, instead of accumulating, according to a given scheme, compound on compound forming even at the present time an almost inextricable tangle? It stands to reason that this study must be based on exact experimental methods.

Has M. Winkler proved that my methods are inexact or false? In no way. Although he claims to have rigorously followed my instructions for the principal reaction, he has in reality modified it, after finding that it was rather difficult to carry out. Now it is necessary to take the trouble to make an intimate mixture of red phosphorus and nitrate of ammonium, and it is also necessary to apply the heat very carefully to reach the temperature given, 180°. But by following M. Winkler's method, that is to say, by throwing the amorphous phosphorus into the fused nitrate of ammonium, a modification of the highest importance is at once made, as it has long been known that phosphorus burns in an atmosphere of protoxide of nitrogen (fused nitrate of ammonium) as readily as in a current of pure oxygen. On the other hand, the experiments of Spring (*Jahresbericht der Chemie*, 1888, 67; *Arch. Pharm.*, 1892, 230, 159) prove that, pressure alone suffices to combine these bodies when very intimately mixed, and, further, that it is by no means unimportant whether these bodies should be mixed in coarse particles or in a finely divided state using the greatest care. Now, M. Winkler simply produced the combustion of phosphorus in protoxide of nitrogen.

He has not checked the other methods I described, and which enabled me to prove the following facts. On heating amorphous phosphorus with peroxide of barium and dilute nitric acid, we get complete oxidation without the least trace of arsenic being formed; by replacing the dilute nitric acid by strong acid, and operating in the presence of peroxide of barium more or less strong, traces of arsenic are formed. But instead of using strong nitric acid, M. Winkler used chlorine. Further, by causing an energetic oxidising agent, such as sodic hydrate or peroxide of hydrogen, to act on amorphous phosphorus, we are right in supposing that the nitrogen of the air intervenes in the formation of arsenic. In the same manner as ordinary phosphorus sets free the nitrogen of the air, amorphous phosphorus liberates it in the presence of sodic hydrate, and the following reaction takes place in the presence of peroxide of hydrogen:—



This conclusion is so much the less a gratuitous assertion as we do not forget that even the easily decomposed nitrogen compounds, such as ammonia, form arsenic in a similar manner with the help of ordinary phosphorus. We also know that in a strongly alkaline solution the nitrogen of the air undergoes a series of transformations, and in this respect even M. Winkler himself has observed the decomposition of protoxide of nitrogen by amorphous phosphorus.

I will now describe some fresh experiments which have shown the possibility of transmuting phosphorus into antimony. These experiments are not yet completely finished, and I shall have more to say on the subject later on, but for the present they warrant me in stating that antimony is also a nitrated compound of phosphorus. Even at the commencement of my experiments with arsenic I sometimes noticed the formation of a small quantity of antimony as well as arsenic, when nitrite of potassium was used as well as nitrate of ammonium, and the temperature was somewhat increased. I therefore made a series of experiments with a view to making sure of the transmutation of phosphorus into antimony, either alone or mixed with arsenic. I soon found, however, that there were innumerable difficulties to be overcome. By placing ordinary phosphorus in contact with nitrite of potassium

and nitrate of ammonium, phosphoric acid alone is formed. A mixture of amorphous phosphorus, nitrate of ammonium, nitrite of potassium, and potassic hydrate, gives rise to a very vigorous reaction; but the yield is so uncertain that sometimes only arsenic is obtained. The same is the case when we replace the hydrate by carbonate of potassium. When, on the other hand, we substitute peroxide of barium for the nitrite of potassium, we obtain a mass which explodes on heating, especially if no potash is present. A mixture of amorphous phosphorus, nitrite of ammonium and saltpetre, also gives rise to an explosive body. By throwing the latter into fused potash, the reaction is less lively though accompanied by luminous phenomena, but in this case also the result is so uncertain that frequently only arsenic is obtained.

An intimate mixture of amorphous phosphorus, nitrite of potassium, and nitrate of ammonium, without the addition of hydrate or carbonate of potassium, bursts into flame instantly, and practically nothing but phosphoric acid is formed. It is for this reason that carbonate of ammonium has been added to this last mixture, when certainly an action took place,—that is to say, a lively reaction occurred at a temperature below the boiling-point of water, but this temperature did not seem to be sufficient to make the change complete. On then heating over a naked flame a vigorous reaction again occurred, but at a temperature above 100° a violent explosion soon took place. To guard against this danger I did the heating on a sand-bath, in a tube traversed by a current of carbonic anhydride and connected to a condenser, and I found that a mixture of amorphous phosphorus, nitrate of ammonium, nitrite of potassium, and carbonate of ammonium, commenced to react at 70° to 75°, and that the reaction became very lively between 90° and 100°. At this point it became calmer, and continued in a tranquil manner to a temperature above 145°, reaching its end at 150° to 155°, with the accompaniment of luminous phenomena or even of explosion. The mass formed at 140° to 145° consists of a brown compound insoluble in water. It contains antimony in quantities easy to detect, as well as phosphorus, and seems to be phosphoretted antimony (*Fahresbericht der Chemie*, 1873, 229). It is probably this latter body that causes the violent decomposition at a slightly elevated temperature. The antimony does not appear to be accompanied by arsenic at the above-mentioned temperature, but in any case this body only occurs in traces.—*Chemiker Zeitung*, 1900, p. 561.

the intervention of cuprous chloride is made use of. 4. Pan processes; and 5. Barrel processes, which are, however, almost obsolete.

The object of Lixivation processes, dealt with in Section III., is to extract the silver in the form of a solution, from which it can be afterwards precipitated by appropriate means. There are practically only two salts of silver which can be used for these processes, the chloride and the sulphate; the former may be dissolved out by brine or decomposed by sodium hyposulphite, with which silver forms a series of soluble double salts, while the sulphate simply requires hot water for its solution. There are three main series of lixiviation processes in which the above characteristics of silver salts are made use of; they are known as the Augustin, Ziervogel, and Patara processes.

The use of ammonia for dissolving silver chloride was actually tried at the Broken Hill mine, but the loss of ammonia was much too great, besides being most inconvenient in other ways.

The smelting of silver ores, to which Section IV. is devoted, together with enough lead-bearing material to carry away the whole of the silver, is practically identical with the blast-furnace smelting of lead ores; but in the absence of lead, matte or speiss may be relied upon to effect a perfect collection of the precious metals. The use of matte has recently undergone remarkable extension through the introduction of practical means for utilising the sulphur in the ore as a source of heat in the modern system called pyretic smelting, and it has the further advantage of permitting a higher degree of concentration without undue loss.

Looked at from the practical point of view, this work has fully achieved its object; the author has produced a first-class book for practical men, and, aided by the masterly editorship of Sir W. C. Roberts-Austen, has collated and arranged his sections and details so as to combine the maximum of information with the minimum of repetition.

The text is adorned with a number of useful illustrations, and is furnished with a copious Index of over fifteen pages, but we are sorry to see a rather long list of errata.

The Scientific Foundations of Analytical Chemistry Treated in an Elementary Manner. By WILHELM OSTWALD, Ph.D., Professor of Chemistry in the University of Leipzig. Translated, with the Author's sanction, by GEORGE MCGOWAN, Ph.D. Second English Edition, translated from the Second German Edition, with further alterations and additions by the Author. London: Macmillan and Co., Ltd. New York: The Macmillan Co. 1900.

ENCOURAGED by the reception of the first edition, which, within three years, has been translated into English, Russian, Hungarian, and Japanese, the author has brought out a second edition, for an English translation of which we are again indebted to Prof. McGowan. The present work is thoroughly revised, and contains many additions. The author says he has "withstood the inclination to enter into greater detail than formerly, in the second part of the book." This is much to be regretted, for the principles involved are of such magnitude and the reasoning so sound that a little more elaboration would be welcome.

We can strongly recommend the book both to advanced chemists and to analytical students, for there is a notorious want of attention to foundation truths in most of the present systems of chemical study, and it is the author's knowledge of this want that called forth the present volume. There is one paragraph in the preface that is so characteristic that we must quote it as giving the key to the whole book:—

"That there is no such thing as a perfectly insoluble substance, and that absolutely exact methods of separation and estimation are an impossibility—remain not merely

NOTICES OF BOOKS.

The Metallurgy of Lead and Silver. By HENRY F. COLLINS, A.R.S.M., A.M.I.C.E., &c. Part II., Silver. Edited by Sir W. C. ROBERTS-AUSTEN, K.C.B., D.C.L., F.R.S. With numerous Illustrations. London: Charles Griffin and Co., Ltd. 1900. Pp. 352.

As in Part I., on Lead, obsolete processes have received only casual consideration, the aim throughout being to consider the subject from the practical standpoint of the working metallurgist.

The book is divided into four principal sections: on Silver and its Ores; Amalgamation processes; Lixivation processes; and Smelting processes. The first Section deals only with the properties of silver and its principal compounds, and enumerates the principal ores of silver.

Section II. is the largest, as all silver ores, however complex, can be treated by amalgamation after a preliminary roasting with salt; but this, while adding to the expense, also causes additional loss by volatilisation, and in most cases, unless the ore can be amalgamated direct, it is better to adopt a lixiviation or smelting process. Amalgamation processes can be divided under five heads: 1. Direct amalgamation. 2. The Mexican or Patio process. 3. The Caso, Fondon, or Tina processes, in which

unknown to the student, but also occur less frequently, I fear, to the mind of the accomplished analyst than is to be desired in the interests of a sound criticism of analytical methods and their results."

The peculiar charm of Prof. Ostwald's writing is in no way lost in the translation, and a study of the volume will help the student to clearly and pleasurably grasp the principles of the science.

Acetylene. A Handbook for the Student and Manufacturer.

By VIVIAN B. LEWES, F.I.C., &c., Professor of Chemistry, Royal Naval College, Greenwich; Chief Superintending Gas Examiner to the Corporation of the City of London, &c. Westminster: Archibald Constable and Co., Ltd. New York: The Macmillan Company. 1900.

CHAPTER I., Part I., gives a clear and comprehensive history of acetylene, from its discovery by Edmund Davy, in 1836, to its commercial production in 1895. The remainder of Part I. is taken up with descriptions of the preparation, properties, and chemical reactions of the gas, recounting much of the detailed communications of those who have recently worked upon the subject. The question of priority of the discovery of the commercial production of calcium carbide is given at length, and includes letters between Mr. Thomas L. Willson and Lord Kelvin upon the subject.

Part II. commences with the Electric Furnace, and gives a complete account of the present manufacture, properties, and impurities of calcium carbide, and the production of acetylene.

Part III. contains the legal enactments in force in various countries, and extracts of English patents for generators and burners, &c.

The whole subject is dealt with in great detail, the book comprising nearly a thousand pages with over two hundred illustrations. There is also an appendix with weights and measures, tables of atomic weights, &c., which seem a little out of place. The book will be of great service to all interested in the manufacture and commercial uses of acetylene.

Les Nouveautés Chimiques pour 1900 (Chemical Novelties for 1900). By M. CAMILLE POULENC. With 182 Figures in the Text. Paris: J. B. Baillière et Fils. 1900. Pp. 292.

This volume will be of great service to chemists, inasmuch that it contains an account of the latest new methods and apparatus introduced for use in commercial chemistry and scientific research.

In the first chapter we find chiefly apparatus designed for the determination of densities, temperatures, solubilities, &c.

The second chapter is principally concerned with apparatus for facilitating and simplifying long and tedious manipulations; it also contains an account of several new types of gas-burners, a new form of mercury pump in which the mercury is raised automatically, designed by M. Berlemont, an apparatus for the manufacture of acetylene black, &c.

The third chapter comprises electrical apparatus of all kinds, including a new electric furnace by Dr. Helbig, some new forms of interruptors, &c.

The fourth chapter deals with general analytical chemistry: among the novelties in this subject will be found a very ingenious apparatus introduced by M. le Docteur, for gas analysis, by means of which the operations are much simplified; there is also to be found a new colorimetric method for the estimation of nitrogen.

Finally, in chapter five, we find the latest improvements in bacteriological methods and apparatus, among which may be mentioned Lequeux's electric sterilising oven.

CORRESPONDENCE.

THE SOCIETY OF CHEMICAL INDUSTRY OF VICTORIA.

To the Editor of the Chemical News.

SIR,—I am instructed by the Council to send you notice of the formation of the above Society.

Enclosed you will find a copy of the circular issued by the Provisional Committee, to which I have made some additions.

The number of members enrolled so far is 118.

The first paper is to be read on September 4th, 1900, by Dr. Avery, M.Sc., on "The Cyanide Process for Gold Extraction."—I am, &c.,

HERBERT W. GEPP, Hon. Sec.

Australian Explosives and Chemical Co.,
Deerpark, Victoria, Australia,
August 27, 1900.

(CIRCULAR).

At a meeting held on the 12th of June at the University, at which between thirty and forty gentlemen representing Chemical and Allied Industries were present, it was unanimously resolved to form a Society to be called "The Society of Chemical Industry of Victoria."

The objects for which the Society is established are:—

- To afford its members opportunities of meeting, and discussing matters connected with Applied and Industrial Chemistry.
- Generally to advance the cause of Chemical Industry in Victoria.

The Society will be open to all who are interested in Industrial Chemistry.

It is proposed that the work of the Society shall be carried on by the following means:—

A meeting will be held once a month during the greater part of the year, at which papers will be read and discussed on some special branch of Industrial Chemistry.

Such meetings will enable members to become personally known to one another, and will encourage the interchange of knowledge and opinions.

Special Committees may be appointed from time to time to report upon questions of interest to those engaged in the Manufacture or Sale of Chemicals, such as want of uniformity in analytical methods or laws affecting Chemical Industries.

Occasionally it may be found advisable to print, for issue to members of the Society, papers that have been read at its meetings, reports of Special Committees, or other matter of general interest. But it is not proposed, at present at all events, to attempt the publication of a journal.

The very great success of the English Society of Chemical Industry, which was established in 1882, and now has over two thousand members on its roll, affords striking proof of the good to be done by such an Association. Much of the work of that Society is done by the meetings of its various local sections, and there can be little doubt that what has proved so great a boon to Chemists in London, Liverpool, Manchester, Nottingham, Glasgow, and other industrial centres at home, will succeed in Melbourne also.

It is indeed certain that the want of such an organisation has been felt in the past, and it is time that this want were met.

I am instructed by the Provisional Committee to invite you to become one of the Original Members of the Society. If you desire to do so, will you kindly fill in and return the enclosed form as soon as you conveniently can.

Notice will then be sent you of the first General Meeting, together with a copy of the laws as drafted by the Provisional Committee. The business of the General

Meeting will include the adoption of the Laws of the Society and the election of office-bearers.

The first annual subscription, which has been fixed at 10s. 6d., may be paid to me at or before that meeting.

HERBERT W. GEPP,
Hon. Secretary of the Provisional Committee.

Provisional Committee:

Professor ORME MASSON, the University of Melbourne (Chairman), elected President.

D. AVERY, Lecturer on Chemistry at the Working Men's College, elected Hon. Treasurer.

J. KERR FORREST, care of Messrs. Felton, Grimwade, and Co.

C. NAPIER HAKE, Chief Inspector of Explosives, elected Vice-President.

WM. KITCHEN, care of J. Kitchen and Sons, South Melbourne, elected Vice-President.

W. A. MILLER, care of Colonial Sugar Refining Co., Yarraville.

A. PARKER, Metallurgical Works, Footscray.

F. STEEL, care of Cuming, Smith and Co., Yarraville.

H. W. GEPP, care of Australian Explosives Co., Ltd. (Hon. Sec.).

The Council elected at the First General Meeting consists of the above, with the following additions:—

JAMES CUMING, jun., care of Cuming, Smith, and Co., to be a Vice-President.

A. N. PEARSON, Chemist to the Departments of Agriculture, Water Supply, &c., of Victoria.

HENRY C. JENKINS, Victorian Government Metallurgist.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 11, September 10, 1900.

Nitrocelluloses.—Léo Vignon.—This paper contains an account of a series of experiments on the reducing power of the nitrogen derivatives of cellulose, and on the creation of these reducing powers by nitration. Two specimens of nitrocellulose and of nitro-oxycellulose were prepared. In the first experiment of each series the nitration was allowed to attain its maximum, in the second a lower nitrated product was obtained. The same operations were performed when oxycellulose (prepared by means of chlorate of potash and hydrochloric acid) was substituted for cellulose. Four products were obtained, in which the author estimates the nitrogen present as a measure of the degree of nitration. He concludes from the numbers deduced—(1) That nitrated celluloses and oxycelluloses energetically reduce cupro-potassic solutions; (2) that their reducing power is independent of the degree of nitration of cellulose or of oxycellulose; (3) the reducing power is almost the same for nitrated cellulose and for nitrated oxycellulose; (4) finally, that the reducing power sensibly constant with regard to nitrocellulose or nitro-oxycellulose, is about one-fifth of that of "invert sugar."

No. 12, September 17, 1900.

Decomposition of Nitric Ethers and Nitroglycerin by Alkalies, and the Relative Stability of Explosive Materials.—M. Berthelot.—In general, ethers are decomposed by alkaline hydrates with formation of the acid and alcohol corresponding to the ether. However, nitric ethers offer certain exceptions to this rule. The author observed that nitric ethers treated with concentrated alkaline solutions can produce methylic and ethylic ethers instead of the corresponding alcohols.

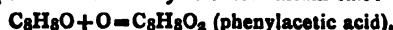
Action of Iodine and Yellow Oxide of Mercury on (1) Styrolene and 2) Safrol.—J. Bougault.—As a continuation of a former research on the action of iodine and yellow mercuric oxide on anethol, isosafrol, &c., the author studies the action of the same reagents on similar compounds, such as styrolene and safrol. The separate reactions obtained when the above substances act on styrolene can be represented by the equations—



The substance thus obtained ($\text{C}_8\text{H}_7\text{I.OH}$) is very easily decomposed by silver nitrate—



This aldehyde, when treated with alkaline silver oxide, gives phenylacetic acid, which can be recognised by its fusing-point and the analysis of its barium salt:—



The reactions are conducted in exactly the same manner with safrol. A compound being obtained by the action of I and of HgO which is an addition product of hypodorous acid; but this body, decomposed by silver nitrate, gives a substance showing none of the characteristic reactions of aldehydes. The author proposes to further investigate this compound.

Reduction of Nitrocelluloses.—Léo Vignon.—This paper is a continuation of that given in *Comptes Rendus*, cxxxi., No. 11, and deals more generally with the reduction of nitrocelluloses. The author first considers the action of ferrous chloride in eliminating the NO_2 groups, but leaving intact the aldehydic groups. The reducing action of ammonium sulphide is more complicated than that of ferrous chloride; it eliminates the NO_2 groups and destroys the aldehydic group CHO. Generally it may be stated that nitrated celluloses reduced by FeCl_2 are transformed into oxycelluloses. Nitrated celluloses, treated with ammonium sulphide, give cellulose or hydrocellulose possessing no reducing power. This difference of action can be explained if we consider that, in the first case, the reduction is effected in an acid medium, and so gives the products of an oxidising reaction ($\text{Fe}_2\text{Cl}_6\text{NO}_2$), whilst, when ammonium sulphide is used, the reaction takes place in an alkaline medium; the products of the reaction, $(\text{NH}_4)_2\text{SO}_4$, NH_4NO_3 , being deprived of their oxidising properties. These results confirm the conclusions previously established, that nitrated celluloses ought to be considered as derivatives of oxycellulose.

Journal de Pharmacie et de Chimie,
Series 6, Vol. xii., No. 1.

Action of Wood Charcoal on the Organic Matter in Water.—F. Malméjac.—Already inserted in full.

Gasometric Estimation of Nitrites in the presence of Nitrates or other Soluble Salts.—J. Gailhat.—Already inserted in full.

Contribution to the Analysis of Sugar Materials.—G. Halphen.—The substance is placed in a flask having the 300 c.c. mark on its neck; such a quantity is taken as will represent about 30 grms. of the total sugars; water is added, and the whole shaken up with an excess of carbonate of lime to saturate the free acids; after ten minutes, neutral acetate of lead is poured into this liquid as long as a precipitate is formed, and 3 or 4 c.c. in excess added. Make up to 300 c.c., shake well, and filter. One portion of this liquid serves for the direct estimation of the reducing sugars; another is examined in the polarimeter; a third is inverted; a fourth, carefully measured (50 c.c.), is evaporated until its volume is only 5 c.c.; it is then carefully heated until dry, heated to 40° , and 0.5 c.c. of pure hydrochloric acid added; after stirring it is precipitated with alcohol. After one to two hours the clear liquid is decanted, and the residue washed with alcohol as

described by M. Raczkowski. Under these conditions the polarimetric estimation can be carried out with great accuracy.

No. 2.

Some Compounds of Diantipyrine-methane (Formopyrine).—G. Patein.—The author took 10 grms. of iodine and 10 grms. of formopyrine and dissolved each of them in 150 to 200 c.c. of alcohol at 95°; the former solution was then poured drop by drop into the latter with constant stirring; fine needles are soon formed, and these increase with each addition of iodine. These crystals have all the appearance of iodine, but they remain stable at a high temperature and do not colour starch paper, they do not melt or give off iodine until a temperature of 135° is reached. Analysis shows this compound to be a tetraiodide of formopyrine. Formopyrine also acts on pyrocatechin, forming a crystalline body corresponding to 4 molecules of pyrocatechin-monosulphonic acid to 5 grms. of formopyrine. Similar bodies are also formed by the action of formopyrine on hydroquinone, and on resorcin.

The use of Dried Pulverised Fibrine for the Estimation of Pepsin.—P. Macquaire.—The author finds that fibrine washed and dried at 40° may be advantageously substituted for fresh fibrine in the ratio of 25 grms. of dry fibrine for 100 grms. of fresh. It permits the use of a 100 c.c. flask and other convenient modifications.

MISCELLANEOUS.

Schools of Chemistry.—The following additional information of interest to Students has been received:—

SURGEONS' HALL, EDINBURGH.—Dr. Stevenson Macadam.—The courses of instruction in Chemistry, Theoretical and Technical, qualify for Graduation in Medicine and in Science in the University of Edinburgh, the University of London, and other Universities, and all other Medical and Public Boards. The Lectures on Chemistry are delivered at 10 a.m. daily during the Winter Session, in the Lecture Room, Surgeons' Hall, Nicolson Street. These Lectures form a complete course, and include Tutorial and Class Examinations. The Preparations on Practical Chemistry are delivered daily during the Winter, Spring, and Summer Sessions. The substances selected for examination and testing are those with which Medical and Pharmaceutical students are personally called upon to exhibit a practical acquaintance. Instructions in Laboratory Work as required for Science (Public Health) Degrees are conducted daily, and include the Testing of Complex Substances, the Detection of Poisons in Organic Mixtures, the Analyses of Air and other Gases, Waters, Foods, and Drugs, &c. The course of study in Technical Analytical Chemistry is arranged for instruction in ordinary Chemical Analyses bearing upon Agriculture, Mining, and Manufacturing Industries, and the prosecution of researches in Manipulative and Technical Chemistry.

Messrs. John J. Griffin and Sons, Ltd., are now introducing a lot of new apparatus for volumetric work. The graduations on the various burettes, pipettes, &c., are made with regard to the fact that 1 kilogram. of water at 15° (the ordinary temperature) is not exactly a litre, but is known as a *Mohr's litre*. The standard litre, as is well known, weighs 1 kilogram. at 4°, and is the basis on which all measuring instruments used by the German Customs' Authorities are graduated. Seeing that at 15° there is a difference of about 2 grms. between the litres of the two systems, it is of the utmost importance, if accuracy in volumetric work be desired, to know on which system the measuring instruments are graduated, and as instruments of both kinds have come into the market it has become imperative to adopt distinctive markings for the two systems. In future all instruments sold by Messrs.

Griffin and Sons on Mohr's system will be marked grm., or grm. grammes
15°C., or 15°C., while those adjusted according to the

standard litre will be marked ccm. or $\frac{\text{ccm.}}{15^\circ\text{C.}}$. These latter vessels hold a standard litre or a cubic decimetre—but not a kilogramme—of water at 15°. At this temperature, and under a pressure of 760 m.m., it weighs only 998.07 grms.; a Mohr's litre under the same conditions weighs 1000 grms.

Volumeiric Estimation of Nickel.—G. Giorgis.—The salts of this metal are totally precipitated in neutral solution by a solution of oxalate of barium or strontium, acidulated with oxalic acid. The precipitate has the formula $\text{C}_2\text{O}_4\text{Ni}$. We can either estimate the oxalic acid contained in the washed precipitate colorimetrically with SO_4H_2 , or estimate the oxalic acid in the excess of oxalate used in the filtered solution. Oxalate of magnesium precipitates a mixed oxalate of nickel and magnesium. The ammoniacal salts ought to be eliminated, as they interfere with the total precipitation of the oxalate.—*Gazz. Chim. Ital.* (1), vol. xxix., p. 72.

Contribution to the Study of Ruthenium and its Compounds.—V. Antony and A. Lucchesi.—The authors draw attention to the fact that no ruthenic compounds of the type RuX_4 are known which do not contain a NO group besides the halogens. They have also examined the blue compounds which are formed by the different ruthenic compounds, and have not yet been isolated. Their experiments have been carried out on the chlororuthenates and sulphate of ruthenium submitted to the action of H_2S and SO_2 . These observations are still incomplete, but full details of what has been done will be found in the original paper.—*Gazz. Chim. Ital.* (1), vol. xxix., p. 312.

The Oxidation of Camphene.—I. L. Maievsky.—On treating a solution of 30 grms. of camphene in 200 grms. of acetic acid, with a 6 per cent solution of permanganate of potash at the ordinary temperature, camphenylone, $\text{C}_9\text{H}_{14}\text{O}$, was obtained, giving an oxime fusible at 104.5° to 105°; the same ketone was found in the products of oxidation of camphene by nitric acid of 1.4 density diluted with its own volume of water, the mixture being heated on the water-bath for eight hours. The oxidation of camphene by CrO_3 in the presence of acetic acid, on the contrary, gives camphor, the oxime of which melts at 114°–114.5°; to effect this change a solution of 30 grms. of camphene in 150 grms. of acetic acid is added to a solution of 60 grms. of CrO_3 in 60 grms. of water and 400 grms. of acetic acid; as the reaction gives off heat the mixture is kept cool at first, and finally heated for a few instants on the water-bath. The author has also found that the aldehyde formed by the dehydration of campheneglycol becomes oxidised when shaken up with the chromic mixture at about 70° to 80°, into an acid, $\text{C}_{10}\text{H}_{16}\text{O}_6$, fusible at 54° to 58.5°, and volatile in steam.—*Fourn. Soc. Phys. Chim. R.*, vol. xxxi., p. 678.

The Ethylenediaminic Compounds of Nickel.—N. S. Korsanof.—When we gradually add ethylenediamine to an aqueous solution of NiCl_2 , the colour passes from green to blue, then to reddish-violet. The blue and violet solutions may be diluted with water without changing the tint; they are distinguished by their reactions as well as by their colours, and they contain complex compounds, such as $\text{NiCl}_2 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2 \cdot m\text{H}_2\text{O}$. On evaporation the violet solution gives violet, rhomboidal flakes, one c.m. in length, having the composition $\text{NiCl}_2 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2 \cdot 3\text{H}_2\text{O}$ stable in the air. The concentrated aqueous solutions of this body are violet, the dilute solutions are a reddish-violet; their reactions indicate the existence of a radical $\text{NiCl}_2 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2$. With PtCl_2K_2 , they immediately give an abundant pale-rose precipitate, formed of microscopic needles, having the composition $\text{NiCl}_2 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2 \cdot \text{PtCl}_2$. With H_2PtCl_6 we obtain a

yellowish-brown flocculent precipitate formed of microscopic needles. Dithionate of soda gives a crystalline precipitate of the composition $\text{NiS}_2\text{O}_3 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2$. A solution of iodine in potassic iodide gives a red precipitate of the polyiodide. The addition of alcohol precipitates the violet salt from its aqueous solutions. The blue solution behaves differently; it has not been obtained in the crystalline form; when evaporated on the water-bath, or in an exsiccator, it leaves an amorphous, viscous mass of a blue colour. Its reactions indicate the presence of a radical $\text{NiCl}_2 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2$, corresponding to the $\text{NiX}_2 \cdot \text{NH}_3$ type of ammoniacal compounds. K_2PtCl_6 gives a precipitate $\text{NiCl}_2 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2 \cdot \text{PtCl}_2$ with the blue solution, formed of small hexagonal prisms of an orange-yellow colour. With the concentrated solution, PtCl_6Na_2 produces a yellowish-orange precipitate, formed of small quadrangular flakes, having the composition $\text{PtCl}_2 \cdot \text{NiCl}_2 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2$. Alcohol and dithionate of soda do not precipitate the blue solutions; the solution of iodine in potassic iodide gives a steel-grey polyiodide. Both the solutions are decomposed by the mineral acids, and by $\text{SO}_2(\text{NH}_4)_2$. Strengthening the tint by the addition of ethylenediamine results in the successive formation of the complex compounds $\text{NiCl}_2 \cdot 2\text{C}_2\text{H}_4(\text{NH}_2)_2$ and $\text{NiCl}_2 \cdot 3\text{C}_2\text{H}_4(\text{NH}_2)_2$. Reciprocally, the addition of NiCl_2 to the violet solution brings the colour back to blue by the inverse reaction—



—*Journ. Soc. Phys. Chem. R.*, vol. xxxi., p. 688.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

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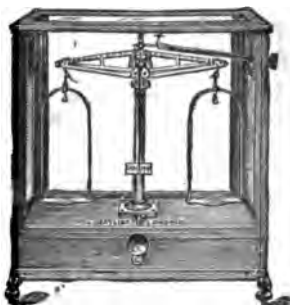
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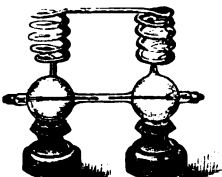
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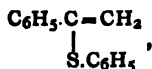
VOL. LXXXII., No. 2133.

ON THE COMBINATION OF THIOPHENOL AND GUAIACOL WITH THE ESTERS OF THE ACIDS OF THE ACETYLENE SERIES.*

By Dr. S. RUHEMANN and H. E. STAPLETON, B.A. (Oxon.).

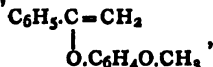
In this paper an account is given of the continuation of previous work on the combination of phenols with the esters of the acetylene acids. The authors were induced to study the action of thiophenol on these esters in the hope of finding a new method of preparing thioacetophenone, whilst the investigation of guaiacol was taken up, as no derivative of a dihydric phenol had previously been worked with.

During the progress of the research various new derivatives of thiophenylcinnamic, fumaric, and succinic acids were discovered, but it was found that thiophenyl styrene,—



on boiling with dilute mineral acids is decomposed into acetophenone and thiophenol, and not into thioacetophenone and phenol, as had been expected.

Guaiacol was found to combine readily with phenyl propiolic ester; guaiacolyl cinnamic acid and its ester were prepared, and it was found that the acid on heating quantitatively decomposed into carbon dioxide and guaiacolyl styrene,—



From the latter mineral acids regenerated guaiacol, with the additional formation of acetophenone.

HEATING AND LIGHTING POWER OF COAL-GAS.*

By T. FAIRLEY, F.R.S.E., F.I.C.

THE consumption of coal-gas for heating and gas-engine purposes is constantly increasing, and in populous districts varies from 20 to 50 per cent of the amount manufactured.

Legislation provides a candle unit for measuring the lighting power of gas, but no notice has been taken of variations in heating power.

As to the relations of heating and lighting power, opinions differ. On one side it is said that a high lighting-power gas is no better than a lower gas for heating and engine purposes (*Encycl. Brit.*, vol. x., p. 102). On the other, a definite relation has been traced between the two (Aguillon, *Comptes Rend.*, cvii., p. 56—58; *J. S. C. I.*, 1894, p. 26).

To obtain the best lighting or heating effects, the apparatus must be adapted to the gas. By ignoring this, either as regards lighting or heating burners or gas-engines, misleading results may be obtained.

From an economic point of view each candle unit costs so much, and it is a question of price whether the extra candle of 16 over 15 candle gas, or of 17 over 16, &c., is worth the additional cost of production. If the heating power marches with the lighting power, the same consideration applies to the heating value.

* Read before the British Association (Section B), Bradford Meeting, 1900.

From a health point of view the less gas that can be made to supply the light required the better. The introduction of the incandescent and other improved burners show how much we have still to learn in obtaining light by the combustion of coal-gas.

The luminosity of the incandescent burner is largely an effect of high temperature rather than of quantity of heat, and I shall not refer to it further in this paper.

Coal-gas is a complex mixture, varying in composition with the coal distilled and the conditions of manufacture. Speaking broadly, it is a mixture of hydrogen and marsh-gas with smaller quantities of heavy hydrocarbons, oxides of carbon, aqueous vapour, nitrogen, &c.

In ordinary burners the lighting power depends chiefly on the heavy hydrocarbons. The heating value is also affected by these, but depends mainly on the marsh-gas and hydrogen, which form from 70 to 80 per cent of the gas.

In gas made from common coal, with a lighting value of 12 to 17 candles, the heating and lighting values march well together. If any material proportion of air is drawn into the gas before it is consumed, or if the gas first given off from the coal is collected separately from that given off last, the relations of heating and lighting powers are materially affected. Air or nitrogen in gas lowers the lighting power more than the heating power, whereas heavy hydrocarbons have an opposite effect. This latter consideration applies to gas enriched with light petroleum ("carburine") or with benzol, and explains why carburetted water-gas has a much lower heating value in proportion than coal-gas. In my own and other experiments its heating value is lower by at least 10 per cent than coal-gas of the same lighting power.

In gas made from the same kind of coal the heating and lighting powers march together, and a calorimeter kept constantly working may be used to watch the gas in place of a jet photometer. With mixed gases it would not be applicable, but in that case only a full photometric test would give the true lighting power.

Since gas has been so much used for heating, the measurement of its heating or calorimetric value assumes additional importance. Various calorimeters have been devised by Berthelot in France, Junker in Germany, and Dowson in this country.

The two latter are adapted for continuous working, and are very similar in principle and construction. In both, the products of combustion are made to pass through metallic syphons, so as to give up all their heat to a circulating current of water. Knowing the quantity of water heated during an experiment by so many degrees, the heat units are obtained, and, reading the volume of gas consumed during the experiment, the heat units per cubic foot of gas are calculated. In these instruments the steam produced by the combustion of the hydrogen in the gas is condensed into water. If it is desired to deduct the heat due to this condensation, the water is collected and measured. With such a large surface wet with the condensed water this correction can only be an approximate one. The amount of water obtained from gas of from 15 to 18 candles averages about 25 c.c. per cubic foot of gas, corresponding to 15 calories, or about 60 British thermal units.

The following average results have been obtained with coal-gas:—

Lighting power, English standard candles.	Heating power, British thermal units. (Pounds of water heated 1° Fahr., not corrected for steam condensed).
11	533
12	555
13	578
14	601
15	624
16	648
17	676
18	704

These numbers are comparable with those of Aguitton, but lower.

To obtain trustworthy results the following precautions are useful:—The calorimeter should be carefully shielded from draughts. The gas should pass through a very efficient governor, and the water supply should be as constant in pressure as possible, and should leave the calorimeter at a constant level, that of the top of the instrument. It is well to place a mirror under the burner so that the flame may be seen at any moment. The flame should be clear and steady, and no result should be accepted with a flickering or unsteady flame.

In starting the instrument it is often some time before a steady flame can be obtained, and for this reason the instructions sent out with the calorimeter, to turn off the gas at the meter at the close of an experiment, could be much improved by directing the gas to be turned into a bye-pass past the meter, so that the working of the calorimeter for the next experiment will not be interfered with.

ISOMORPHOUS DERIVATIVES OF BENZENE.*

THE existence of morphotropic relationships between the crystalline forms of substances which are not isomorphous in the formal sense of the term has of recent years acquired new importance, the purely geometrical work of Barlow (*Proc. Roy. Soc.*, 1897, viii., 527) and others having demonstrated the superfluity of the old view that the units of the crystalline structure are polymerides of the chemically active fundamental molecule as a means of explaining polymorphism and kindred crystallographic phenomena, whilst Fock (*Zeit. f. Kryst.*, 1897, xxviii., 337) has shown, from the study of the partition coefficients of two isomorphous substances in equilibrium in a liquid and a solid solution in contact, that in the case of salts, at all events, the molecular weight in the crystalline state may be that of the fundamental molecule. Moreover, the work of Paternò (*Gazzetta*, 1895, xxv., i., 411) and others on the cryoscopic behaviour of substances possessing constitutions similar to that of the solvent, indicates with certainty that isomorphism and morphotropy are phenomena which merge gradually one into the other.

The consideration of facts such as these leads to the conclusion that morphotropy and isomorphism have a common cause, and that this is more likely to be discovered by the crystallographic study of substances showing morphotropic relationships than from the examination merely of materials likely to exhibit isomorphism.

The benzene series offers exceptional opportunities for the study of such questions; indeed, it is remarkable that the publication of Groth's important memoir (*Pogg. Ann.*, 1870, cxli., 31), calling attention to the existence of morphotropic relationships between benzene derivatives, has not acted as an incentive to really systematic work on the subject.

The two investigations to be referred to form part of a series which are being carried on in the chemical department of the Central Technical College, South Kensington, in order, as far as possible, to determine the effect on the crystalline form of certain definite changes in the composition. The work will include the determination of the molecular volumes and of the melting-point curves of mixtures of the morphotropically related compounds.

Morphotropic Relationships between Formanilide and its Substitution Derivatives.

In order to determine the morphotropic effect of substitution upon the crystalline form of formanilide the simple anilides of the composition $C_6H_5.NH(CO.X)$

(X=H, Me, Et, Pr, &c.) as well as several of their alkyl-derivatives, and also a number of the mono- and di-halogen derivatives, have been crystallographically examined by Mr. L. P. Wilson; a list of the compounds measured is given in Table I., in which the geometrical constants are also indicated. It is evident that a progressive change in the structural dimensions occurs as each series is traversed, although in most cases this only becomes obvious on re-arranging the axial ratios, and sometimes on taking simple multiples of the ratios. The form in which the axial ratios are compared is indicated in the second column.

In Series I., although the first member is monosymmetric, whilst the others are orthorhombic, a well-marked morphotropic similarity in the magnitudes of the ratio a/b is observed to follow the displacement of the hydrogen atom in the acidic group by Me, Et, or Pr. The effect of displacing the aminic hydrogen atom by Me, Et, or Pr is less, as is shown by an inspection of Series II.; in this case the ratio c/b is more nearly constant throughout the series than is the case in Series I. In Series III., obtained by displacing the acidic hydrogen atom in parabromformanilide by either Me or Et, the ratio a/b again shows approximate constancy. No simple relationship is observable between parabromacetanilide and its methyl or ethyl derivative. Parabrom- and paraiod-acetanilide are isomorphous; the corresponding chloro-derivative is not isomorphous with them, although it bears a marked morphotropic relationship to them (compare Fels, "Dissert.," Leipzig, 1900). Group 6 forms a well-marked isomorphous series.

Otten (*Zeit. f. Kryst.*, xvii., 391) has observed that butyranilide is dimorphous, but has not examined the substance in great detail; a study of this compound has shown that the dimorphism is of a very remarkable character. At ordinary temperatures the anilide separates from alcoholic solutions in large transparent crystals of pyramidal habit, which are distinctly orthorhombic, showing a characteristically orthorhombic interference figure of small optic axial angle. The axial ratios of such crystals are $a : b : c = 0.6920 : 1 : 0.6792$. On preserving crystals which had been measured at a constant temperature of 8° to 11° they have been found to change gradually, and in the course of three months completely, into tetragonal crystals, without at the same time losing their brilliancy and transparency. The axial ratio $a : c = 0.6652 : 1$ in these crystals; they exhibit the characteristic uniaxial interference figure. On preserving the definitely tetragonal material at 30° for eighty days the reverse change occurs, the crystals becoming orthorhombic and biaxial, although the axial ratios never revert to quite their original values. The density of the orthorhombic form is 1.130 , whilst that of the tetragonal form is 1.139 .

The molecular volumes of several of the anilides have been determined, with the object of examining the relations between the optic axial ratios of Muthmann (*Zeit. f. Kryst.*, 1894, xxii., 497); the ordinary axial ratios seem, however, in most cases to express the morphotropic relationships just as clearly as the optic ratios.

Iso- and Polymorphous substituted Benzene-sulphonic Chlorides and Bromides.

It has already been stated (*B. A. Report*, 1899, p. 688) that the sulphonic chlorides and bromides derived from the 1 : 3 : 4 dihalogen-benzene-sulphonic acids together form an isomorphous series. Dr. Jee's further study of this group has led to important results. The series includes anorthic, orthorhombic, and monosymmetric terms, in the manner shown in Table II.

Of these eight substances, three are stable in the anorthic system, three in the orthorhombic system, and two in the monosymmetric system. Change of the one form into the other has been observed in four cases (IV., V., VII., VIII.) on allowing the fused substance to cool on a microscopic slide; the direction of the change in each case is indicated in the table by an arrow. A

* Report of the Committee, consisting of Professor H. A. Miers (Chairman), Dr. W. F. Wynne, and Dr. H. E. Armstrong (Secretary). Drawn up by the Secretary. Read before the British Association (Section B), Bradford Meeting, 1900.

TABLE I.

I.										β	
Formanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 2.188 : 1 : 2.403$	90 54 M
Acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 2.0670 : 1 : 0.8488$	90 O
Propionanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 2.1665 : 1 : 1.0428$	90 O
Butyranilide	..	..	..	..	..	..	..	..	..	$3a : b : c = 2.1663 : 1 : 1.3788$	90 O
II.											
Acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 2.0670 : 1 : 0.8488$	90 O
Methylacetanilide	..	..	..	..	..	..	..	..	..	$2c : b : a = 0.7906 : 1 : 0.8494$	90 O
Ethylacetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.0064 : 1 : 0.8401$	90 O
Propylacetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.3264 : 1 : 0.8410$	90 O
III.											
P.-brom.-formanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.4100 : 1 : 2.2056$	90 O
„ acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.3904 : 1 : 0.7159$	90 O
„ propionanilide	..	..	..	..	..	..	..	..	..	$3a : b : c = 1.3400 : 1 : 0.8948$	90 O
IV.											
P.-brom.-acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.3904 : 1 : 0.7159$	90 O
„ methyl acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.5546 : 1 : 0.9719$	70 7 M
„ ethyl „	..	..	..	..	..	..	..	..	..	$a : b : c = 1.4063 : 1 : 1.5686$	95 35 M
V.											
P.-chlor.-acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 1.3263 : 1 : 0.6804$	90 O
P.-brom- „	..	..	..	..	..	..	..	..	..	$a : b : c = 1.3904 : 1 : 0.7159$	90 M
P.-iodo- „	..	..	..	..	..	..	..	..	..	$a : b : c = 1.4185 : 1 : 0.7415$	90 29 M
VI.											
2 : 4-Dichlor.-acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 0.8263 : 1 : 0.6828$	77 33 M
„ Chlorbrom.-acetanilide	..	..	..	..	..	..	..	..	..	$a : b : c = 0.8144 : 1 : 0.6722$	77 40 M
„ Bromchlor- „	..	..	..	..	..	..	..	..	..	$a : b : c = 0.8214 : 1 : 0.7074$	77 46 M
„ Dibrom- „	..	..	..	..	..	..	..	..	..	$a : b : c = 0.8131 : 1 : 0.6895$	78 24 M

TABLE II.

	Orientation.			Crystallographic systems.		
	1.	2.	3.	Anorthic.	Orthorhombic.	Monosymmetric.
I.	Cl	Cl	SO ₂ Br	Stable	—	—
II.	Cl	Br	SO ₂ Br	Stable	—	—
III.	Br	Cl	SO ₂ Br	Stable	—	—
IV.	Br	Br	SO ₂ Br	Labile→	Stable	—
V.	Br	Br	SO ₂ Cl	(Labile)→	Stable	Labile
VI.	Br	Cl	SO ₂ Cl	—	Stable	Labile
VII.	Cl	Br	SO ₂ Cl	—	Labile→	Stable
VIII.	Cl	Cl	SO ₂ Cl	—	Labile→	Stable

labile anorthic crystalline form of dibromo-benzene-sulphobromide (IV.) has been obtained from solution ; and it has been found that each of the four sulphochlorides (V.—VIII.) can be caused to crystallise in the alternative system by admixture with a sulphochloride which usually separates in that system. It has been possible to determine the symmetry of all the forms referred to in the table by crystallographic measurement, with the single exception of the labile anorthic form of dibromo-benzene-sulphochloride, but the existence of this form is indicated by the dimorphous change which occurs on cooling from the melting-point to the atmospheric temperature.

The detailed study of such a series of isomorphs—especially of the melting-points of mixtures and of the conditions which determine the separation of the various crystalline forms—will be of importance, as it is likely to furnish information of value in discussing the phenomena presented by igneous rocks containing isomorphous minerals, and by alloys.

The investigation has been extended by Dr. Jee to the corresponding derivatives of the 1 : 3 : 5 dihalogen-benzene-sulphonic acids. The results obtained indicate the existence of an isodimorphous series having no apparent similarity with the 1 : 3 : 4 series. One of the members of this new series—1 : 3 : 5 dibromo-benzene-sulpho-

bromide—has been obtained in two distinct crystallographic forms, both belonging to the monosymmetric system. At atmospheric temperature one of these forms is labile and isomorphous with the corresponding sulphochloride, and with 1 : 3 : 5 bromo-chloro-benzene-sulphochloride. The stable form of 1 : 3 : 5 dibromo-benzene-sulphobromide, on the other hand, is isomorphous with 1 : 3 : 5 bromo-chlorobenzene-sulphobromide. The derivatives of the symmetrical dichloro-acid have not yet been satisfactorily measured.

Even in their present incomplete form these results are of considerable importance as showing the manner in which the occurrence of polymorphism may render obscure otherwise well-marked isomorphous or morphotropic relationships. Apparently a substance may crystallise in a whole series of different forms, A, B, C, D, the particular form obtained under ordinary conditions being the form stable at the temperature at which the crystals are grown. Another substance, the immediate homologue of the first in an isomorphous series, can also assume crystalline forms corresponding with A, B, C, D, &c., but the particular form stable at ordinary temperatures will not be the same as before, owing to the non-correspondence of the transition temperatures. Consequently the first member of an isomorphous series may crystallise in a

form of type A, the second member in a form of type B, the third of type C, and so on, the isopolymorphism completely masking the isomorphism.

RECENT RESEARCHES ON INDICAN.

By EDWARD SCHUNCK, Ph.D., D.Sc., F.R.S.

RECENT researches, chiefly by Dutch chemists, have thrown much light on a very interesting question, that of the state in which the colouring-matter of indigo exists in the cells of plants. Since the date of the publication of my researches (1855-56) very little attention had been paid to the subject, this being in a great measure due to the difficulties of the investigation, difficulties of which I myself had some experience, and which were reflected in the imperfect nature of my results. It was therefore with no small interest that I read the memoir of Prof. Hoogewerff and Mr. Ter Meulen, in which the authors describe their method of preparing a colourless crystalline glucoside in place of the amorphous indican, the only compound yielding indigo hitherto known (*Kon. Akad. v. Wetenschappen*, April, 1900). Though the existence of such a compound had been anticipated by Dr. Marchlewski and Mr. Ratcliffe, still its actual preparation was a meritorious achievement.

Mr. Hazewinkel has also published a memoir in the same journal as the preceding, in which he shows that indican, by decomposition with acids, enzymes, &c., yields indoxyl, which by subsequent oxidation is converted into indigo-blue. This might have been anticipated, since it was shown several years ago, by Dr. Roemer and myself, that indican in watery solution does not yield indigo-blue with acid, unless an oxidising agent, such as ferric chloride, is present at the same time.

Prof. Hoogewerff, of Delft, had the kindness to forward to me a small specimen of his indican, sufficient to enable me to confirm his account of its properties. My examination, however, though of course very slight, led to the conclusion either that my indican must have been very impure even for such a substance, or that there exists more than one substance to which the name has been applied. Assuming the last supposition to be correct, and giving the name *alpha-indican* to the original substance as described by me, and that of *beta-indican* to the substance of the Dutch chemists, I may indicate in a few words the distinguishing characteristics of the two as follows:—

1. Alpha-indican and beta-indican are soluble in water, alcohol, and ether.
2. The solutions of alpha-indican are coloured yellow on the addition of alkalis, and give yellow precipitates with lead acetate, while the solutions of beta-indican remain white with alkalis and give white precipitates with lead acetate.
3. By the action of strong acids and a mild oxidising agent alpha-indican yields indigo-blue, indigo-red, and a body which resembles but is not identical with glucose, inasmuch as it is not fermentable, while beta-indican with acid and ferric chloride is simply decomposed into indigo-blue and glucose.
4. By the action of caustic alkalis at a boiling temperature alpha-indican is changed and modified in some way, so as now to give with acids very little or no indigo-blue, but in its place indigo-red, whereas beta-indican, if I understand aright what Prof. Hoogewerff and Mr. Ter Meulen say, is not affected by the action of caustic alkali.

Anyone conversant with this department of Chemistry, and reading the description of the two bodies without prejudice, would probably come to the conclusion that they are distinct, not mere modifications of one substance. Of course it is easy to say that beta-indican represents

the pure substance deprived of the impurities accompanying it in the original substance; this is possible, and even probable, but not proven. To have obtained a comparatively stable crystallised substance in place of what previously represented it, which was amorphous and ill-defined, is a very meritorious piece of work, but it does not exhaust the question.

Anyone reading my early memoirs on this subject would see how carefully I avoided, in the preparation of indican, the use of an elevated temperature, even that of boiling alcohol. The extraction of the material, and the subsequent evaporation of the extracts, were made in the cold, because I convinced myself that, at a higher than ordinary temperature, indican, especially in watery solution, is modified so as no longer to yield indigo-blue on treatment. On the other hand, turning to the memoir of Messrs. Hoogewerff and Ter Meulen, we find them, in the preparation of their indican, employing boiling water, boiling alcohol, and even baryta water, all potent agents very likely to lead to metamorphosis in any body liable to change. In my opinion, therefore, the indican of Messrs. Hoogewerff and Ter Meulen is a derivative of alpha-indican or some similar substance, due to the action on it of agents apparently only employed for the purpose of purification. If I am asked how it is possible for a stable body like beta-indican to be derived from a very unstable body by the action of potent agents, I can only say I have no answer to give. I can only point to analogical cases, and of these I may mention that of chlorophyll. This body, when in solution, is very susceptible to the action of air and light, being rapidly bleached and decomposed thereby, but when its solutions are treated for even a short time with sulphuric or hydrochloric acid, products of decomposition are formed which are remarkably stable, resisting the action of air and light not for days only, but for weeks, and even months. The action of alkalis on chlorophyll leads, in a similar manner, to the formation of products of great stability. I have met with similar instances in other departments, such as that of the glucoside of madder, which, as my experiments prove, does not pre-exist as such in the plant; but I will not enter further on this at present. These facts have been strenuously denied by observers who choose to shut their eyes, but there can be no doubt of their reality for all that. And so in the case of indican as in that of chlorophyll, there may exist, before final decomposition takes place, intermediate products of which the final resultant products do not lead us to suspect the existence.

Prof. Beijerinck has thrown some doubt on the existence of indican in the leaves of *Isatis tinctoria*, and prefers to attribute the indigo-producing properties of woad to the presence of a body called by him *isatin*, which he has not isolated, and which, by the action of an enzyme called *isatase* present along with it in the plant, yields indoxyl, and then, by oxidation, indigo-blue. I confess I am at a loss to understand this view. Indican was first obtained by me from woad leaves, and the properties of the substance subsequently obtained from *Polygonum tinctorium* seeming to be precisely the same, I concluded the two were identical. I have repeated lately Prof. Beijerinck's experiment of passing air through an extract of woad leaves with boiling water, but no indigo-blue was precipitated until either a strong acid or an alkali was added, showing that if indoxyl was present in the extract it was certainly not in a free state.

In confirmation of this view, that woad leaves contain indoxyl either free or combined, Prof. Beijerinck states that if isatin be added to a watery extract of woad leaves, and the liquid be boiled, indirubin, or at all events a substance dissolving in alcohol with a bright red colour, is deposited. This does not, however, always take place. Sometimes the deposit does not consist of indirubin, but of a peculiar substance which I discovered many years ago on adding isatin to a boiling extract of woad leaves, though I am not sure that I have anywhere described it. This substance, when pure, appears in micro-

scopic crystals, and in mass is dark blue, almost black. It might be mistaken for indigo-blue were it not for the peculiar bronze-like (not coppery) lustre which it exhibits when rubbed with a hard body. It may be kept for years in a dry state, but if its alcoholic solution, which is bright blue, be left to stand only a few minutes, the colour changes to yellow, and the substance is completely metamorphosed. In acting on indican with acids some indirubin is always formed, but much more when the indican has been previously acted on by a caustic alkali, as I have pointed out on previous occasions.

The discordant results obtained by Dr. Marchlewski in his examination of indirubin would almost lead one to infer that there are several substances of similar properties comprised under the head of indirubin, from whatever source derived. The decomposition of indican, whether it be a glucoside or not, so as to yield indigo-blue as one of its products, has been ascribed by some to the action of microbes, by others to that of a peculiar enzyme. There is one process, however, observed by me which cannot, I think, be ascribed to either cause. It is as follows:—If a plant of *Polygonum* or of *Isatis* is immersed in a vessel of water, and the vessel is placed in a freezing mixture until the liquid has become one mass of ice, it will be found, after thawing, that the leaves that were thoroughly frozen will be found limp, flaccid, and discoloured, and, after treatment with boiling or even cold alcohol to remove the chlorophyll, those very parts will appear blue, sometimes a very dark blue, while the leaves and parts of leaves not affected by the excessive cold will be almost colourless. That the indigo-producing body of leaves should be changed in the same way, by exposure to unusual cold, as by the action of acids, alkalis, or ferments, is indeed strange, and cannot, in my opinion, be explained in any of the usual ways. Bacteria would seem in this case to be excluded, as it is difficult to conceive the possibility of their existing and acting within a block of ice. It may be surmised, too, that any plant-enzyme or enzymes would fare no better as agents of decomposition. No fermentation of any kind can take place at a temperature of 0°: this is conceded by all who have discussed the subject of fermentation. Some other cause must be found for the peculiar effect of cold which takes place equally in the case of *Isatis* and *Polygonum*, and must in both cases be the same. The same effect as by cold is produced by dipping the leaves of either plant into moderately strong alcohol, which on standing removes the chlorophyll, the leaves remaining more or less deeply coloured blue. This reaction has already been described by Prof. Beijerinck, but he has not, in my opinion, sufficiently explained it. I will not enter into speculations on this matter, as these would be more or less hypothetical.

Kewal, Manchester, October 10, 1900.

NOTES ON THE FERROCYANIDE TITRATION OF ZINC.*

By EDMUND H. MILLER and E. J. HALL.

THE experiments recorded in this article were undertaken to find out the influence of hydrochloric acid and those salts most likely to be present, on the estimation of zinc by titration with potassium ferrocyanide, using uranium acetate as an indicator.

The solutions used were:—

1. Potassium ferrocyanide, 43.2 grms. of the crystallised salt per litre.
2. Zinc chloride, 12 grms. (approximately) per litre, containing 40 c.c. of hydrochloric acid, 1.2 specific gravity.
3. Uranium acetate, a saturated aqueous solution.

* Contribution from the Havemeyer Laboratories, Columbia University. From the *School of Mines Quarterly*, xxi., No. 3.

The method and conditions of titration were as follows:—50 c.c. of the zinc chloride solution, containing 2 c.c. of hydrochloric acid, were measured into a beaker by a pipette; the salt, or acid, whose influence was to be tested, added; then water to bring the total volume to 200 c.c. The solution was then heated to boiling, and, after stirring vigorously, titrated. The conditions for titration, so far as volume and temperature are concerned, were in all cases 200 c.c. and 80°—90° C. The end point was determined in the usual way, using drops of uranium acetate on either sized paper, or on a porcelain tile. A light but distinct brownish flesh colour was taken as the end. The allowance necessary to turn the indicator in the presence of a c.c. of hydrochloric acid was found to be 0.3 c.c. on paper and 0.35 c.c. on porcelain. This difference is one of individual eyesight, and is given to explain why the quantities subtracted are not uniform.

Effect of Hydrochloric Acid.

C.c.	C.c.
2 HCl 1.2 sp. gr. required	0.35 K ₄ Fe(CN) ₆ solution.
4 " " "	0.45 " "
6 " " "	0.60 " "
8 " " "	0.75 " "
10 " " "	0.85 " "

Each result is the average of three determinations made on porcelain. More hydrochloric acid exerts such a solvent action on the uranium ferrocyanide as to render the indicator unreliable.

C.c.	C.c.	C.c.
50 ZnCl ₂ solution containing 2 HCl required	27.65	
50 " " "	4 " "	27.70
50 " " "	6 " "	27.80
50 " " "	8 " "	27.90
50 " " "	10 " "	28.00
50 " " "	12 " "	28.10
50 " " "	14 " "	28.20

Correcting by blank test we get that 50 c.c. of the zinc chloride solution containing 2 c.c. of hydrochloric acid requires 27.3 c.c. of the ferrocyanide solution. This is the result of six determinations, and is the value used throughout. The other results are averages of two or three tests. By subtracting 27.3 from the values given we get the amounts required for the indicator in the presence of zinc to be, for 4 c.c. HCl, 0.4 c.c.; 6 c.c. HCl, 0.5 c.c.; 8 c.c. HCl, 0.6 c.c.; 10 c.c. HCl, 0.7 c.c.; 12 c.c. HCl, 0.8 c.c.; 14 c.c. HCl, 0.9 c.c. Determinations made on paper gave slightly lower results: 6 c.c. HCl, 0.45 c.c.; 8 c.c. HCl, 0.5 c.c.; 10 c.c. HCl, 0.6 c.c.

These results show the great influence of hydrochloric acid on the indicator, and that it is diminished by the presence of zinc ferrocyanide.

Effect of Magnesium Sulphate.

Moldenhauer (*Chem. Zeit.*, 1892, No. 14) states that in the titration of zinc by potassium ferrocyanide in an ammoniacal solution magnesium interferes, as the ferrocyanide is not readily soluble in ammonia. The following tests were made to ascertain whether it had any influence in an acid solution. To 50 c.c. of the zinc chloride solution containing 2 c.c. of hydrochloric acid, different quantities of MgO as sulphate were added, and the solution titrated under the standard conditions.

0.01 gm. MgO required	27.65 c.c., corrected	27.35 c.c.
0.10 " " "	27.70 " "	27.40 " "
1.00 " " "	27.70 " "	27.40 " "

In order to test the solvent effect of magnesium sulphate on the indicator, two portions of crystallised magnesium sulphate, 6.111 grms. each (1 gm. MgO), were dissolved and titrated in the presence of 2 c.c. of HCl. They each required 0.6 c.c.

Assuming that the solvent action is less in the presence of zinc, as is the case with hydrochloric acid, we conclude that the interference of magnesium is negligible in an acid solution, and is due to a slight solvent action on the indicator, not to the formation of magnesium ferrocyanide.

Effect of Calcium Chloride.

The following weights of calcium chloride were added to portions of zinc chloride solution as described under magnesium sulphate:—

0.05 gm. CaCl_2 required	27.65 c.c., corrected	27.35 c.c.
0.50 " " "	27.85 " "	27.55 "
5.00 " " "	28.00 " "	27.70 "

Our experiments accord with the statement by Low (*Journ. Am. Chem. Soc.*, 1893, p. 552), that calcium acetate exerts a solvent action on uranium ferrocyanide, and so interferes in the lead titration by potassium ferrocyanide.

Effect of Ammonium Chloride.

As small amounts of iron are usually separated from zinc, in the analysis of ores, by the ammonia in the presence of a large amount of ammonium chloride, the effect of this salt is of importance. To 50 c.c. of zinc chloride solution containing 2 c.c. of hydrochloric acid, the following weights of ammonium chloride were added before titration under the usual conditions:—

5 grms. NH_4Cl required	27.85 c.c., corrected	27.55 c.c.
10 " " "	27.90 " "	27.60 "
20 " " "	28.00 " "	27.70 "

Tests were next made to see whether the error was due to a solvent action on the indicator. Ten grms. NH_4Cl added to 2 c.c. of HCl and titrated as usual gave a sharp end-point at 0.3 c.c.; 20 grms., the same result, 0.3 c.c., showing that the effect of large quantities of ammonium chloride was to sharpen the end-point (compare Beringer, "Text-book of Assaying," p. 221). That the influence of ammonium chloride is on the zinc ferrocyanide and not on the indicator is shown by the marked difference in the appearance of the solution titrated when ammonium chloride is present. The precipitate is greenish white and flocculent at the beginning of the titration; near the end, when about 0.3 c.c. more is required, as indicated by uranium acetate, there is a sudden change—the precipitate becomes brownish white and the green colour entirely disappears.

To confirm our results the following tests were made:—

NH_4Cl solution.	ZnCl ₂ solution.	HCl.	Change in precipitate.	Vol. used.	Corrected.
	C.c.	C.c.	C.c.	C.c.	C.c.
0	50	2	None	27.65	27.30
5	50	2	27.5	27.70	27.40
10	50	2	27.4 (about)	27.80	27.50
15	50	2	27.5	27.70	27.40
20	50	2	27.6	27.85	27.55

This change apparently coincides with the complete conversion of the zinc to $\text{K}_2\text{Zn}_2\text{Fe}_2(\text{CN})_{12}$. It does not take place when the titration is made in an ammoniacal solution, where the normal zinc ferrocyanide, $\text{Zn}_2\text{Fe}(\text{CN})_6$, is formed.

Effect of Aluminium Sulphate.

The removal of copper from zinc ores can be readily effected by aluminium foil in a sulphuric acid solution (Furman, "Manual of Practical Assaying," p. 209), but the uncertainty of the end-reaction with uranium acetate suggested the following experiments:—To 50 c.c. portions of the zinc chloride solution containing 2 c.c. of HCl, 0.20 gm. of Al_2O_3 was added in the form of crystallised aluminium sulphate. The amount of hydrochloric acid was varied.

Al_2O_3 .	HCl.	Volume used.	Corrected.
	C.c.	C.c.	C.c.
0.20	2	22.80	22.50
0.20	3	22.30	21.95
0.20	5	26.80	26.40
0.20	7	28.40	27.95
0.20	7	27.75	27.35
0.20	7	28.60	28.15
0.20	7	28.00	27.55

The influence of aluminium sulphate is very marked, giving low results with small amounts of acid, and in all cases irregular results. In the presence of this salt it is impossible to ascertain the end-point with certainty, as the brown colour appears very gradually and is never distinct.

Experiments were made with no zinc present which gave with 2 c.c. and with 6 c.c. of HCl, 0.7 c.c. as the allowance for the indicator, but the colour was not satisfactory.

When potassium ferrocyanide is added to a neutral solution of aluminium sulphate, the aluminium being in excess, and boiled, a white precipitate is formed, similar in appearance to zinc ferrocyanide; a drop of the solution containing this precipitate gives a brown colour with uranium acetate. When hydrochloric acid is added to the solution containing the precipitate, the reaction with uranium acetate is checked. A precipitate is formed on heating a solution of aluminium sulphate with potassium ferrocyanide, even in the presence of considerable hydrochloric acid.

The interference of aluminium with the indicator seems analogous to that of manganese (Stone, *Journ. Am. Chem. Soc.*, 1895, xv., p. 473) and is sufficiently marked to render its use for removing copper very objectionable.

Effect of Lead Chloride.

If we discard the use of aluminium to precipitate copper, we must go back to lead as the best substitute. The following experiments were made to determine how much hydrochloric acid is necessary to prevent any precipitation of lead ferrocyanide. The titrations were performed under the usual conditions, the lead being added as chloride to the zinc solution.

Pb.	HCl.	Volume required.	Corrected.
Grm.	C.c.	C.c.	C.c.
0.005	2	27.70	27.40
0.01	2	27.75	27.45
0.02	2	27.80	27.50
0.04	2	27.90	27.60
0.08	2	28.00	27.70
0.16	2	28.05	27.75
0.32	2	29.60	29.30
0.64	2	37.10	36.80

Two c.c. of hydrochloric acid is insufficient for more than a trace of lead.

Pb.	HCl.	Volume required.	Corrected.
Grm.	C.c.	C.c.	C.c.
0.32	4	27.90	27.55
0.64	4	29.00	28.65
0.32	6	27.85	27.40
0.64	6	27.90	27.45
0.32	8	27.95	27.45
0.64	8	27.95	27.45
0.32	10	28.00	27.40
0.64	10	28.05	27.45

This shows that 6 c.c. of hydrochloric acid, 1.2 sp. gr., will prevent any interference due to lead. As Comey's "Dictionary of Solubilities" states that lead ferrocyanide is soluble in sodium citrate, we tried this salt to prevent the precipitation of the lead, but found that, while it prevented the precipitation of lead, it had such a solvent action on uranium ferrocyanide that it could not be used. Citric acid was also tried without satisfactory results.

Effect of Bismuth Nitrate.

Small quantities of bismuth as nitrate were added to the zinc chloride solution and titrated as usual.

Bi. Grm.	HCl. C.c.	Volume required. C.c.	Corrected. C.c.
0.005	2	27.80	27.50
0.01	12	28.00	27.40
0.02	12	28.00	27.40
0.04	12	28.00	27.40

Small amounts of bismuth have no influence.

Effect of Antimony Trichloride.

0.10 grm. of antimony trichloride was added to the zinc chloride with sufficient acid to prevent the precipitation of basic salts.

SbCl ₃ . Grm.	HCl. C.c.	Volume required. C.c.	Corrected. C.c.
0.10	8	28.2	27.7
0.10	10	28.4	27.8

Antimony interferes with the titration.

We conclude from these experiments :—

1. That the amount to be subtracted, as shown by a blank test, is often greater than the excess required in the actual titration.
2. That salts (such as calcium chloride, sodium citrate) and acids (notably hydrochloric) retard the end-reaction by their solvent action on uranium ferrocyanide.
3. That ammonium chloride, not exceeding 10 grms., does not interfere with the accuracy of the method, but has some (unknown) effect on the zinc ferrocyanide.
4. That the presence of considerable quantities of aluminium sulphate—such as would be present if copper were removed by aluminium foil—renders the results unreliable.
5. That 6 c.c. of concentrated hydrochloric acid is sufficient to prevent any interference by lead.
6. That antimony gives high results, while small quantities of bismuth have no influence.

THE FORMULA OF COBALT PEROXIDE.

By THOMAS BAYLEY, Assoc.R.C.Sc.

A PAPER by Mr. T. Moore on the separation of nickel and cobalt appeared in the CHEMICAL NEWS of August 17th (vol. lxxxii., p. 73), in which he referred to the formula of the peroxide of cobalt precipitated by means of hypochlorites, hypobromites, &c., and incidentally mentioned the formula Co_3O_5 assigned by myself to the peroxide of cobalt in 1879 (*Proc. R. I. A., Journ. Chem. Soc.*).

The method by which the composition of this oxide, and of Ni_3O_5 , was determined was similar to that used by Mr. Moore. namely, dissolution of the washed peroxide in a liquid containing an acid and potassium iodide and titration of the liberated iodine with thiosulphate. But since the majority of dictionaries and text-books have not adopted the formulae Co_3O_5 and Ni_3O_5 , in spite of subsequent confirmation of my results by other chemists, it occurred to me, about two or three years ago, to re-determine the composition of cobalt peroxide by a somewhat different method, and Mr. Moore's paper having recalled attention to the matter, the opportunity seems favourable for publication of the results so obtained.

The cobalt was weighed in the form of sulphate after careful ignition to low redness, which I have found to be one of the best forms in which to weigh both this metal and nickel. After precipitation, the peroxide was washed on an asbestos filter, and then dissolved in dilute sulphuric acid in presence of a known excess of ferrous sulphate, and the amount of Fe remaining unoxidised determined with standard potassium bichromate.

The general result of these experiments seems to be that, at ordinary temperatures, both chlorine and bromine in presence of caustic soda precipitate an oxide of the form Co_3O_5 , and that at 100°C . a lower—and perhaps rather less definite—form (which may be Carnot's $\text{Co}_{20}\text{O}_{16}$) is produced. The action of peroxide of hydrogen at the ordinary temperature is much less definite. The writing of the various formulae in terms of Co_{60} is merely for purposes of convenient comparison.

$\text{CoSO}_4 = 464$ Grms. was taken for each Experiment.

	Fe oxidised. Grm.	Formula indicated.	Conditions of experiment.
1.	0.1855	$\text{Co}_{60}\text{O}_{93.2}$ $\text{Co}_{60}\text{O}_{93} = 9\text{CoO}, 11\text{CoO}_2$ requires 0.1841 grm.	NaHO and NaClO ; and mixture heated to 100°C . (boiled).
2.	0.1832	$\text{Co}_{60}\text{O}_{93.8}$ Do.	Do.
3.	0.2258	$\text{Co}_{60}\text{O}_{100.4}$ $\text{Co}_{60}\text{O}_{100} = \text{Co}_3\text{O}_5$ requires 0.2232 grm.	NaHO and NaClO at ordinary temperature = 15°C .
4.	0.2243	$\text{Co}_{60}\text{O}_{100.2}$ Do.	Do.
5.	0.2218	$\text{Co}_{60}\text{O}_{99.7}$ Do.	NaHO and NaBrO at ordinary temperature.
6.	0.2172	$\text{Co}_{60}\text{O}_{98.9}$ Do.	Do.
7.	0.1347	$\text{Co}_{60}\text{O}_{84.1}$ $\text{Co}_{60}\text{O}_{84} = \text{Co}_5\text{O}_7$ requires 0.1339 grm.	$\text{NaHO} + \text{H}_2\text{O}_2$ at ordinary temperature.
8.	0.1109	$\text{Co}_{60}\text{O}_{79.9}$ $\text{Co}_{60}\text{O}_{80} = \text{Co}_5\text{O}_4$ requires 0.1116 grm.	Do.
9.	0.1878	$\text{Co}_{60}\text{O}_{93.6}$ $\text{Co}_{60}\text{O}_{93} = 9\text{CoO}, 11\text{CoO}_2$ requires 0.1841 grm.	$\text{NaHO} + \text{NaBrO}$, and mixture heated to 100°C . (boiled).
10.	0.1931	$\text{Co}_{60}\text{O}_{94.6}$ $\text{Co}_{60}\text{O}_{95} = \text{Co}_{12}\text{O}_{19}$ requires 0.1953 grm.	Do.; but scarcely raised to boiling.
11.	0.1897		NaHO and NaBrO , after boiling for thirty minutes.
12.	0.0942		AmHO and H_2O_2 , boiling.

The separation of the precipitated Co_3O_3 (hydrated) by filtration seems to be quite unnecessary; even in the cold the excess of hypochlorite being quickly decomposed by catalytic action, with evolution of oxygen; and if, after this has ceased to come off, iodide of potassium be added, followed by excess of hydrochloric acid, and the liberated iodine be titrated with thiosulphate and starch indicator, a measure of the cobalt is obtained.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART IV. NICKEL AND COBALT.

By HARRY BREARLEY.

(Continued from p. 43).

The arrangement of items is similar to that of the previous parts, i.e., Separations, Estimations, Detection, and Miscellaneous Notes, and journals to end of 1899.

NICKEL.

I. SEPARATION OF IRON.

1. *By means of Acetates.*—The separation of nickel from iron by means of acetate requires greater manipulative precision than the like separation of any other element of the same group, and therefore a few references to papers which discuss the basis of acetate separations are included.

493. MACKINTOSH (C. N., lvi., 65).—Acetate separation needs to be repeated six times. Cobalt more difficult to separate than nickel. Precipitates Ni, Co, and Fe as sulphides, treats precipitate with HCl, and makes acetate separation of the soluble and insoluble portions.
494. WESTERSON (Y. I. S. I., 1894, ii., 495).—Sample dissolved in H_2SO_4 , oxidised, and Fe repeatedly precipitated. Mn interferes with electro-deposition of Ni.
495. BREARLEY (C. N., lxxiv., 16).—Precipitation from a solution containing 7 per cent acetic acid. Ni titrated with KCN and AgI; copper separated as sub-sulphocyanide; SO_2 and KCNS do not interfere with titration of the Ni.
496. DEBRAY (C. N., xxi., 53).—Ferric acetate separates on boiling into acetic acid and colloidal oxide; the latter is insoluble in ammonium salts and acetic acid.
497. BREARLEY (C. N., lxxvi., 222, &c.).—Ease of separation of the following elements is in the order Mn, Zn, Co, Ni. Attempts to deduce reasons for this from the character of the salts separated. Separation of various elements without a second precipitation (C. N., lxxvi., 49 and 222, Nickel; lxxvi., 165, Cobalt).
498. GIBBS (C. N., xi., 102).—Add enough acetate to convert all bases into neutral acetates to the cold solution.
499. BREARLEY (C. N., lxxix., 193).—It is best, according to both theory and practice, to add acetate to the boiling solution. (See also 1, 3, 4, and 8).

1. 2. *Precipitating Iron as Hydrate.*—

500. THOMAS (C. N., xxxv., 187).—Repeated precipitation of the Fe with excess of ammonia.
501. SLEEPER (C. N., lxxix., 15).—Decompose material with aqua regia, KBr and Br, or NaHSO_4 . Precipitate Ni and Fe with NaHO, dissolve in H_2SO_4 , and rapidly add $(\text{NH}_4)\text{HO}$ to the cold solution. Detailed instructions for electro-deposition of the Ni.
502. NEUMANN (Y. I. S. I., 1898, ii., 560).—The Fe is separated with $(\text{NH}_4)\text{HO}$ and $(\text{NH}_4)_2\text{SO}_4$, and the Ni electro-deposited from the filtrate.

503. MOORE (C. N., xlv., 76).—To faintly acid solution add $(\text{NH}_4)_2\text{SO}_4$ and large excess oxalic acid; precipitate Fe with $(\text{NH}_4)\text{HO}$ (see 525).
504. EMMENS (Y. I. S. I., 1893, i., 414).—Precipitated ferric hydrate boiled with dilute acid; only little Fe dissolves along with the Ni; Ni and Co are electro-deposited. Applied to regulus, minerals, and alloys (Y. S. C. I., 1892, 1035).
505. JUPTNER (Y. I. S. I., 1894, i., 616).—Precipitate with $(\text{NH}_4)\text{HO}$ in the presence of $(\text{NH}_4)\text{Cl}$, and keep hot several hours. (Repeated precipitation is necessary, Y. I. S. I., 1894, ii., 495). Ni and Co precipitated as sulphides, and Mn and Al separated.
506. MOORE (C. N., lxxv., 75).—The separation by NH_4HO^* is worthless; by acetate needs four precipitations. (See also 10, 14, 15, 19a, 21, 22, 23, 26).
507. BLOXAM (C. N., lii., 109).—The precipitation of Fe with $(\text{NH}_4)\text{HO}$ always carries down the Co in larger proportion than the Ni, so much so that Ni may in this way be roughly detected in the presence of cobalt.
508. REINITZER (C. N., xlviii., 114).—Solutions of Cr_2O_3 , when boiled with soda acetate are not precipitated by alkalis, alkaline carbonates, phosphates, &c., and the same property is conferred on certain amounts of Fe or Al present therewith.
- RECOURA (C. N., lxxviii., 46).—The presence of alkaline sulphates have a like effect.

1. 3. *By Precipitating the Nickel.*—

509. THOMPSON (C. N., vii., 185).—To acidulated sulphates of Ni, Co, Fe, Mn, Zn, and Cu add $(\text{NH}_4)_2\text{SO}_4$; nearly all the Ni and Co separate as "alums" on standing.
510. MOORE (C. N., lvi., 3).—Add pyrophosphate until precipitate begins to re-dissolve; add excess KCN, boil, add KHO, and precipitate Ni with KHO and Br. Co remains with the iron; Ni estimated electrolytically. A modified process leaving out pyrophosphate and embracing other separations (C. N., lvii., 125).
511. CAMPBELL and ANDREW (Y. I. S. I., 1895, ii., 598).—Add soda pyrophosphate and soda carbonate; precipitate Ni with soda xanthate; convert to sulphate and electro-deposit, or titrate with KCN and AgI.
512. PINERUA (C. N., lxxv., 193).—Based on the insolubility of the chlorides of Ni and Al and the solubility of the chlorides of Co and Fe in etherised HCl are processes for the separation of Ni and Al from Fe and Co (see 623 and 526).
513. PERILLON (Y. I. S. I., 1898, ii., 562).—Precipitate the dissolved sample with KHO, add oxalic acid, dry at 80° ; boil dried mass with a dilute acetic acid and alcohol, and ignite the precipitated oxalate to NiO .
514. CLASSEN (C. N., xxxvi., 40).—In Wöhler's process for separating As and Ni (544), if Fe is present the Co and Ni are completely precipitated on neutralising, adding acetic acid, and potassium oxalate (see 205).

1. 4. *Electrolytic Separations.*—

515. LE ROY (C. N., lxiii., 194) and HERRENSCHMIDT and CAPELLE (C. N., lxxix., 142).—Ni, Co, and Fe are deposited from a solution containing citric acid, ammonia, and ammonium sulphate; on reversing the current in an ammoniacal solution of $(\text{NH}_4)_2\text{SO}_4$, Fe is precipitated; Ni and Co are deposited together.

* In a private communication dated November, 1898, Moore speaks very highly of a separation with $(\text{NH}_4)\text{HO}$:—"I add an excess $(\text{NH}_4)\text{Cl}$ to the solution, then dilute to about 400 c.c. and carefully add (from a burette) dilute ammonia (1:15) until the Fe_2O_3 , &c., is precipitated. At this stage the solution is very distinctly acid, and the iron at least is perfectly thrown down. . . . I can vouch for its exactness for our (New Caledonian) Co and Ni ores."

516. DUCRU (C. N., lxxvi., 279).—Electrolyse ammoniacal solution containing precipitated iron. A small amount of iron is deposited with the Ni. Mn, P, and large amounts of Cr_2O_3 do not interfere. CrO_3 prevents electro-deposition entirely.
517. NEUMANN (J. S. C. I., 1898, 1074).—On electrolysis as above (516) the deposited Fe varies with the amount in solution, time, strength of current, &c.
518. ENGELS (J. C. S., lxxiv., ii., 192).—The deposited Ni is free from iron if the latter has been oxidised with H_2O_2 . (See also Foerster, J. C. S., lxxiv., ii., 228).
519. VORTMANN (J. C. S., lxxvi., 34).—Zn, Co, and Fe are deposited free from Ni in an alkaline tartrate solution. Ni, Co, and Cu are estimated in presence of Fe, and after the manner of 516.
- I. 5. Miscellaneous Processes.—
520. MOORE (C. N., liv., 300).—Add soda phosphate, clear with acid, boil, and precipitate with acetate; precipitate Ni with KHO, and electro-deposit from sulphates. Shortcomings of Field's (22), Wöhler's (hydrate), and acetate processes enumerated.
521. CHENEY and RICHARDS (C. N., xxvii., 161).—Acetate and hydrate inferior to Field's process. Precipitate with soda phosphate from acetic solution; Ni electro-deposited; with more than 3 per cent Ni, precipitation to be repeated.
522. EASTWICK (J. I. S. I., 1894, i., 615).—Depends on solubility of nickel sulphide and insolubility of iron sulphide in acid solution of $(\text{NH}_4)_2\text{SO}_4$. A little Fe goes with the nickel.
523. GALBRAITH (J. S. C. I., 1882, 345).—Precipitated with calcium sulphide; the Fe dissolved out with HCl; Ni weighed as sulphate.
524. ROSENBLATT (J. C. S., i., 492).—Ni and Co are separated from Fe, Cr, Al, Mn, and Zn by precipitating the latter elements with potassium thiocarbonate.
525. ZIEGLER (J. S. C. I., 1893, 377).—An acid solution of the metals is poured into an alkaline solution of ammonium borate. Ni, Co, and Cu are in solution; Fe, Al, and Mn are precipitated.
526. ROTHE (C. N., lxxv., 182).—Hydrochloric acid solutions of ferric salts are soluble in ether; similar solutions of NiO , MnO , FeO , Cr_2O_3 , and Al_2O_3 are not; hence a means of separating them.
527. BREARLEY (C. N., lxxviii., 15).—Uses chromates instead of acetates. A large excess of the precipitant used in acetic solutions without any Ni being precipitated. Also separates Fe and Cu. (See also 10, 19a, 21, 22, 23, 25).

(To be continued).

PROCEEDINGS OF SOCIETIES.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

October 2nd, 1900.

Prof. HORACE LAMB, F.R.S., President, in the Chair.

MR. WILLIAM BURTON, F.C.S., gave a paper dealing with the question of "Plumbism" among pottery workers, from the technical point of view, showing how the plumbism was caused by the inhalation or swallowing of dust containing soluble lead compounds. It was pointed out that the abolition of lead from the glaze was not a practicable remedy, as leadless glazes suited to the conditions of the general run of English pottery are not yet within the reach of the manufacturers. Further, the abolition of lead from the glaze would not affect those cases which arise from the use of enamel or on-glaze colour used in

the form of dust or of spray, and no one has yet ventured to suggest that the use of lead fluxes for this purpose could be abolished.

The existing remedies in the shape of mechanical means for dealing with the dust in such a way that it should neither be swallowed nor inhaled, the periodic medical examination of the workers, and the proposed further safeguards of converting all the lead used into compounds of considerably lower solubility than those now employed, were also treated of.

The final opinion expressed by Mr. Burton was that the combination of all these safeguards would within a reasonable time render the operations of glazing and decorating pottery with substances containing lead compounds as free from risk to the operative as it was possible for any industrial occupation to be.

NOTICES OF BOOKS.

Fluorine and its Compounds ("Le fluor et ses Composés"). By M. HENRI MOISSAN, Member of the Institute. Paris: G. Steinheil. 1900. Pp. 397.

In this volume the author has collected his numerous papers on fluorine and its compounds which have been published from time to time. Fluorine is a remarkable element, and has been too little studied; too great reliance has been placed on the parallelism of reactions, and the supposed analogy that new compounds of fluorine ought to have with the compounds of chlorine, bromine, and iodine. Fluorine, however, has special properties of its own which, though leaving it at the head of the halogen group, give it a place apart on account of the peculiarity of some of its characteristics.

At the commencement of his research the author directed all his efforts to the isolation of fluorine, and was thus enabled later on to have the command of large quantities of this gas.

New investigations then followed, such as the determination of some of the physical constants of fluorine, combinations with the metalloids and the metals, as well as organic compounds of fluorine.

In his recent investigations the author found it necessary to considerably modify the original form of his apparatus for producing pure fluorine. Its preparation is best effected by means of the electrolysis of hydrofluoric acid in a platinum U-tube, plunged into a vessel containing chloride of methyl; by this means the temperature is kept down to -23° . Owing, however, to the costliness of the platinum apparatus experiments were made with the object of replacing this metal by copper, and very good returns were obtained, though it was found necessary to retain platinum as the material from which the electrodes were made.

The general scheme adopted in carrying out the experiments with fluorine was as follows:—The bodies subjected to treatment with fluorine were divided into three classes—solid, liquid, and gaseous. When a solid body is to be treated a morsel may be placed in the delivery tube of the generating apparatus, or in the cover of a platinum crucible held in the stream of gas. When the substance under examination is a liquid, the fluorine may be simply passed through a glass tube in which the liquid is contained; the glass will not be readily attacked as it becomes protected by a thin layer of alkaline fluoride when the tube is dry, and if wet the moisture protects it; in some cases, however, when hydrofluoric acid is formed during the reaction, platinum tubes should be used. For examining closely the action of fluorine on a gas a special piece of apparatus has been devised; this is fully described and illustrated on pp. 82–83.

The author has again given the world a striking example of his patience and skill; his powers of descriptive writing

are of a high order, while his style and choice of language further help to stamp him as a *savant* of the first rank.

Tonometry ("La Tonometrie"). By F. M. RAOULT, Corresponding Member of the Institute, &c., Scientia Collection, No. 8. Paris: C. Naud. 1900. Pp. 116.

At the present day it is no longer possible for scientific men to become or remain specialists; it is imperative that they should be familiar with the ever growing extensions of the various branches of science; mathematicians, physicians, chemists, and biologists are all becoming more and more closely related, and have more and more interests in common.

The subject of the present monograph, "Tonometry," deals with the vapour tensions of liquid mixtures, of which one constituent at least is volatile; it is closely allied to cryoscopy, both having for their object the study of solutions.

The author first deals with solutions of organic substances or non-electrolytes, while in the second part he deals with solutions of mineral salts or electrolytes. There are several different methods of observation described, such as the static method, and the hygrometric, volumetric, and gravimetric methods; in the author's opinion, the method by boiling is, without question, the best of them all.

The book is handy in form, concisely written, and is decidedly worth reading.

CORRESPONDENCE.

THE PROPOSED NEW "SOCIETY OF CHEMICAL INDUSTRY OF VICTORIA."

To the Editor of the Chemical News.

SIR,—As a member of the Society of Chemical Industry, which Mr. H. W. Gepp, the Hon. Sec. of the Provisional Committee of the Victoria Society, terms the "English Society," may I be allowed to point out that the fourth paragraph from bottom of column 2, page 168, of your issue of October 5th, contains, to my thinking, no less than four somewhat misleading statements. These may be at once enumerated, and also corrected, as follows:—

1. Our Society is not, I think, an "English" Society merely; it is not merely a British Society; it is an Anglo-American Society. Its President of last session was an American, and I believe we are proud of both these facts.
2. Our Society was not established in 1882, but in 1881.
3. As to the Membership roll, the figure given—"over two thousand members"—would be more fairly expressed as "nearly four thousand members."
4. Membership of our Society has not only "proved a boon to chemists in London, Liverpool, Manchester, Nottingham, Glasgow," but also in the United States of America, and it seems somewhat strange that Mr. Gepp in his Circular should omit our most prosperous and rising Section, next to that of London, viz., the New York Section of the Society of Chemical Industry.

He probably would not have made the omission, however, had he been present at our Annual General Meeting this summer, and witnessed the enthusiastic reception accorded to our American President, and have heard his Address, and especially his speech at the Annual Dinner of the Society.

If there is one thing we of the Society of Chemical Industry are more proud of than another, it is of the fact that in the Society and its journal, the hands of British and American technical chemists are united—spite of the broad Atlantic—in a brotherly grip.—I am, &c.,

VERAX.

October 6, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Journal de Pharmacie et de Chimie,
Series 6, Vol. xii., No. 3.

Action of Nitric Acid on Trichlorised Gaiacol.—H. Cousin.—10 grms. of trichlorised gaiacol were dissolved in 50 c.c. of glacial acetic acid, and a mixture of 10 c.c. of nitric and 10 c.c. of acetic acid gradually added. The solution takes a brown colour, gives off reddish fumes, and after a time deposits a bright red crystalline powder. This product is purified by crystallisation in warm ether, and concentration of the ethereal solution. It is insoluble in water, soluble in alcohol, ether, acetic acid, and chloroform, especially when warmed; its fusing point appears to be between 158° and 162°. Analysis leads to the formula $C_{13}H_3Cl_3O_4$. The author then describes its behaviour with benzene.

Identity of *Bacillus Lactis Aerogenes* and the *Pneumobacillus* of Friedländer.—MM. L. Grimbert and G. Legros.—The authors find that both biologically and morphologically, as well as in their action on carbohydrates these bacilli are identical, and they are therefore of the opinion that one name should suffice for both. Their principal characteristics are:—(1) Immobility; (2) the presence of capsules in the blood of inoculated animals; (3) the non-liquefaction of gelatin; (4) the non-production of indol; (5) the energetic action on the carbo-hydrates, giving rise to products variable with the nature of the sugar employed.

Estimation of Uric Acid.—A. Bellocq.—250 c.c. of urine are treated with an excess of soda and filtered; 200 c.c. of the clear filtrate are treated with 20 c.c. of a reagent consisting of 30 c.c. of 33 per cent pure sulphate of zinc, 30 c.c. of soda lye, and 40 c.c. of saturated carbonate of soda. A voluminous precipitate is formed and filtered off. This precipitate is dried in a crucible, and 2 or 3 c.c. of HCl (saturated with pure uric acid) added and kept cool; the crystals of uric acid soon sink to the bottom; these are removed by filtering through cotton-wool, dried, and weighed. The success of the operation depends on the first filtration.

Some Properties of Schinoxydase.—J. Sarthou.—The solubility of this substance is never complete, the filtration of its solution is very slow, and its reaction neutral. When evaporated to dryness it leaves a carbonaceous residue, which, when burnt with nitric acid and treated with water acidulated with hydrochloric acid, gives the reactions of salts of iron. The author describes an experiment showing that this metal exists in the organic state. Schinoxydase is soluble in acids, but all acids do not act in the same manner; traces of a mineral acid destroy the reactions of oxidation, which are slightly retarded by the presence of a small quantity of organic acids, though quite destroyed by a larger dose.

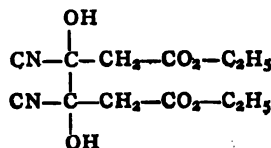
Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 11.

The Ammonio-earthly Phosphates.—L. Barthe.—Already inserted in full.

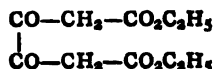
α,β -Trimethyl- β -Oxyadipic Acid.—E. E. Blaise.—Already noticed.

Condensation of Cetipic Ether with the Orthodiamines. 1. Condensation with Ethylenediamine and the Naphthaleneorthodiamines.—R. Thomas-Mamert and St. Weil.—A very long paper, not suitable for abstraction.

Action of Hydrocyanic Acid on Cetipic Ether.—R. Thomas-Mamert and St. Weil.—The experiments here described were undertaken with the object of obtaining the dicyanhydrines of the formula—



by the fixation of two molecules of hydrocyanic acid on cetipic or oxaldiacetic ether,—



This fixation is not effected without considerable difficulty. After several unsuccessful trials it was found best to dissolve 1 molecule of cetipic ether in absolute alcohol, and then add a little more than 2 molecules of pure pulverised cyanide of potassium to the solution. To this mixture, cooled to 0°, the theoretical quantity of concentrated pure hydrochloric acid was added, drop by drop, with constant stirring. After some days a large quantity of a badly crystallised yellow body was formed: when no more cetipic ether is left, which is known by a small quantity evaporated to dryness giving no red reaction with perchloride of iron and a little alcohol, the solution is filtered. The ethereal filtrate on distillation leaves an oil from which fine crystals are obtained; these crystals, dried with blotting-paper and re-crystallised in alcohol, form large white plates, fusible at 164°, giving no reaction with perchloride of iron. This is the dicyanhydrine sought for.

New Colouring-Materials with an Acid Function.—M. Prud'homme.—The leucobase or chromogene colouring-materials, containing NH₂ groups, are treated in acid solution by a mixture of formic aldehyde and bisulphite of soda. The author gives the results of experiments with fuchsine, α-nitrodiaminotriphenylmethane, thionine, safranin, and nitraniline. He finds as he expected, that the mixture of CH₂O and SO₃NaH is without action on the colouring-matters of which the aminogenes are alcoylised, and also on the imidised bodies.

New Blue Colouring-Material, Solid with Alkali, and of Special Constitution.—M. Prud'homme.—This new colouring-matter contains a SO₃H group in the ortho-position with regard to the central carbon; but instead of being connected directly with the benzenic radical, the SO₃H is connected to this radical by the intermediary of the NH.CH₂ group.

Rhodinol and Citronellol.—L. Bouveault.—A controversial paper on the constitutional formulæ of these bodies.

The Transformation of Rhodinal into Menthone.—L. Bouveault.—Not suitable for abstraction.

Research on the Migrations and Metamorphoses of the Terpenic Compounds in Peppermint.—E. Charabot.—As a result of his observations, the author finds that menthol, which is formed at the commencement of vegetation, becomes partially etherified in the leaves; then when buds, and later flowers, are formed, a certain quantity of essence accumulates, and the menthol—both in the combined and free state—is converted into menthone by oxidation.

Influence of Active Vegetation on the Formation of Thuyone and Thuyol.—E. Charabot.—During the period of active vegetation not only the weight of the plant increases considerably, but the relative proportion of essential oil becomes almost double. A notable quantity of thuyol has been formed. This thuyol is trans-

formed partially into ethers, and partially into thuyone by oxidation.

The Presence of Cystine and Tyrosine in Contaminated Waters.—H. Causse.—Already noticed.

MISCELLANEOUS.

Merck's Digest, No. 9.—Stypticin.—This substance is a derivative of the opium alkaloid narcotine, obtained through the action of oxidising agents. The preparation forms a crystalline powder of the colour of sulphur; it dissolves freely in water, giving a straw-coloured solution; its formula C₁₂H₁₄NO₃Cl. In its chemical characteristics stypticin is closely allied to hydrastinin, which it also resembles in its physiological properties. Besides its powerful action in the stopping of bleeding, stypticin has, according to several medical authorities, a well-marked and potent sedative action, so that it is in every way to be preferred to the *extractum hydrastis liquidum*, and even to hydrastinin itself.

Experimental Research on New Explosive and Detonating Materials.—V. Alvisi.—This research has been carried out on the use of perchlorate of ammonium, mixed either with detonating gelatines, gelatinised gun-cottons, or even with mixtures of carbon and sulphur. The author has observed that for similar mixtures the explosion produces its maximum effect when the detonator itself contains perchlorate of ammonia mixed with the fulminate. Finally, the author has endeavoured to solve the problem of the commercial preparation of perchlorate. He has observed experimentally that the substitution of perchlorate of ammonia for the other oxidising salts in the actual explosives, or its addition to the substances which have no need of supplementary oxygen for total combustion, is capable of forming such mixtures as will give simultaneously (1) an increase in the ballistic force, (2) an increase in the disruptive force, and (3) an increase in the proportion between one and two; that is to say, mixtures which have the most advantageous properties, as mine explosives. The mixtures of perchlorate, sulphur, and carbon are made in various proportions; that of 5:5:1:1:3 appears to be one of the most useful. These mixtures have been named *mantianite*.—*Gazz. Chim. Ital.*, vol. xxxix., p. 121.

Some Double Thiocyanates of Vanadium.—A. Cioci.—*Thiocyanate of Vanadium and Potassium*,—Vd(CNS)₃.3KCNS.4H₂O.

This body is obtained by saturating a solution of SO₃H₂ and VdO₃ with SO₂ gas. After driving off the excess of SO₂, the whole is reduced in a negative cell until no further green precipitate is produced with potash. The calculated quantity of thiocyanate is then added, and the product formed, insoluble sulphate of potassium, separated by means of alcohol. It consists of red crystals having a variegated appearance by reflected light, losing 4H₂O at 100° and decomposing at about 110°. The solution gives a precipitate of Vd(OH)₃ with the alkalis, the alkaline earths, and their carbonates. A black precipitate is formed with CuSO₄, a yellowish-red one with NO₃Ag, and a white one with Pb.(C₂H₃O₂)₂. *Thiocyanate of Vanadium and Ammonium*, Vd(CNS)₃.3NH₄CNS.4H₂O.—This body is obtained in a similar manner to the preceding one. It occurs in dark green crystals forming a blood-red powder. It behaves like the last compound. *Thiocyanate of Vanadium and Sodium*, Vd(CNS)₃.3NaCNS.12H₂O.—This body appears in greyish-red crystals, fusible at 68° in their own water of crystallisation. *Chromothiocyanate of Sodium*, CrNa₃(CNS)₆.12H₂O.—This body is obtained from sulphate of chromium and thiocyanate of sodium. It occurs in the form of bright red crystals, losing their water of crystallisation at 100°.—*Gazz. Chim. Ital.* (1), xxix., p. 300.

Action of Peroxide of Hydrogen on the Fatty Amines.—L. Mamlock and R. Wolfenstein.—In reacting with peroxide of hydrogen on dipropylamine the authors have obtained dipropylhydroxylamine, $(C_3H_7)_2N.OH$, a white crystallised compound possessing an agreeable odour; on reduction it re-forms dipropylamine. In the same way as hydroxylamine gives sulphaminic acid when treated with sulphurous acid, so does dipropylhydroxylamine give dipropylsulphaminic acid. This acid turns litmus red; it does not contain either sulphurous or sulphuric acid, and corresponds to the formula $(C_3H_7)_2N.H.SO_3H$. When tripropylamine reacts with peroxide of hydrogen, oxide of tripropylamine is formed quantitatively. This oxide is deliquescent, and possesses basic properties. Its picrate, fusible at 130° , has been analysed. When the oxide is heated two reactions take place; on the one hand there is formation of dipropylhydroxylamine and propylene, and on the other hand regeneration of tripropylamine. When heated in vacuo this second reaction does not take place. The transformation of the oxide of tripropylamine into dipropylhydroxylamine has a certain interest in this sense, that this latter being easily reduced to dipropylamine, we have passed from a tertiary amine of the fatty series to a secondary amine.—*Berichte*, vol. xxxiii., p. 159.

MEETINGS FOR THE WEEK.

WEDNESDAY, 17th.—Microscopical, 8. "Report on the Recent Foraminifera of the Malay Archipelago," Part IX. At 7.30, Exhibition of Slides and Models of Skin Structure, by F. W. Watson Baker, F.R.M.S.

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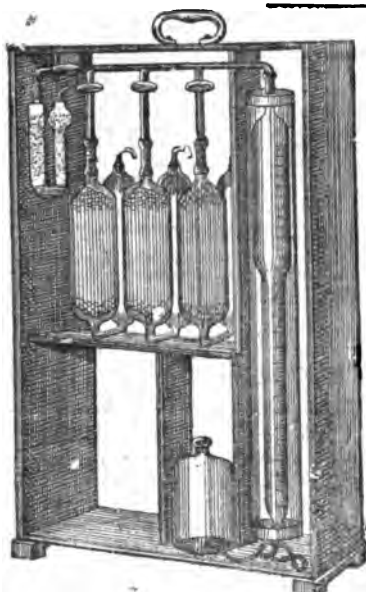
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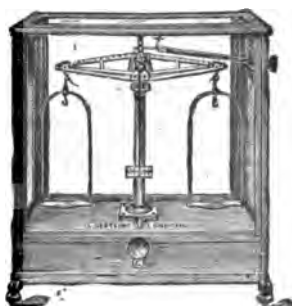
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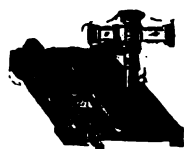
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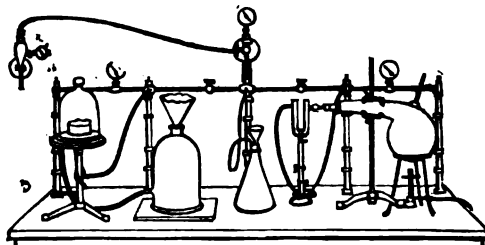
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THE CHEMICAL NEWS.

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AN IMPROVED DESICCATOR AND STIRRING-ROD.

By EDWIN DOWZARD, F.C.S.

EVERYONE has noticed, after placing a hot crucible in the desiccator, the jumping and side-slip of the lid; this is, of course, due to the expansion of the contained air, brought about by the hot crucible; when the crucible has cooled there is a slight vacuum, which renders the lid somewhat difficult to remove; when the lid has been removed there is a sudden inrush of air, which does not improve the efficiency of the desiccator.

Some desiccators have taps which may be opened and shut to allow for expansion and contraction, but the effect is the same, except that the lid does not slip and there is no difficulty in removing it; then there is the trouble of opening and shutting the tap every time the desiccator is used.

The following illustration (Fig. 1) shows how, in a very simple manner, the above faults may be remedied. The glass tube, A, is bent to fit inside the desiccator; the perforated plate, B, is raised sufficiently to allow the tube to pass underneath. It will be seen that this tube allows the ex-

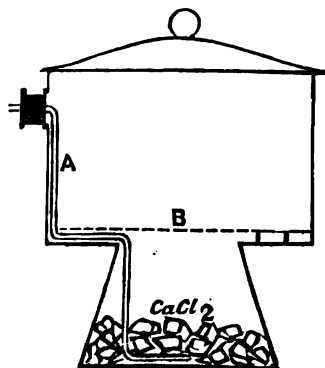


FIG. 1.

panded air to escape, and also allows air to enter, which has, however, to pass through and over the CaCl₂ contained in the lower part of the desiccator, thus ensuring a thorough drying.

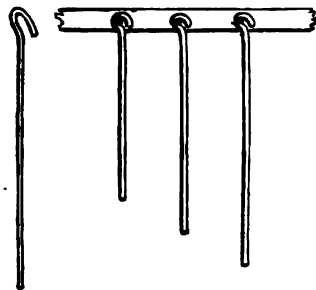


FIG. 2.

FIG. 3.

Fig. 2 represents an ordinary piece of glass tubing, sealed at both ends, one end being bent into a hook. By this means the rod may be suspended from a small nail

(see Fig. 3): the hook also acts as a handle when in use. The stirring-rod is thus always at hand, and the size needed can be selected without any trouble.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART IV.

NICKEL AND COBALT.

By HARRY BREARLEY.

(Continued from p. 181).

NICKEL (*continued*).

II. SEPARATING OTHER ELEMENTS FROM NICKEL.

- Cobalt*.—(See 598, &c.).
528. *Zinc*.—BRUNNER (C. N., i., 95).—With H₂S from solution containing soda acetate. Excess acetate and heat to be avoided. Ni and Co in solution.
529. DOHLER (J. C. S., lxxv., 811).—Precipitate Zn with H₂S in the presence of soda formate and formic acid.
530. GIBBS (C. N., xi., 149).—Add HCN gas and Na₂S to solution containing acetate, or precipitate hot solution with Na₂S and dissolve out Zn with HCl. The former process also separates Ur from Ni and Co.
531. MOORE (C. N., i., 151).—Precipitate with (NH₄)₂S, dissolve in KCN, and precipitate ZnS with soda acetate and acetic acid.
532. VON BERG (C. N., lv., 236).—Precipitated with H₂S from formic or monochloroacetic solutions.
533. HAMPE (J. C. S., lii., 182).—As 532. Monochloroacetic acid the more efficient, especially with Co.
534. BAUBIGNY (C. N., lix., 88).—From acetic solutions with H₂S in the cold. Not available for cobalt (J. C. S., lvi., 653).
535. BEILSTEIN (C. N., xxxix., 74).—H₂S passed into cold ammoniacal solution made acid with citric; Zn precipitated. Electro deposition of Ni made from solution of nitrates; (NH₄)Cl interferes.
536. ALT and SCHULZE (C. N., lxi., 124).—With H₂S from hot or cold solutions of succinic acid. Process for analysing nickel-silver. (See also 524, 573, and 574).
537. *Zinc and Copper*.—CARNOT (C. N., liii., 172 and 196).—Cu precipitated with hyposulphite. Zn from oxalic acid solutions with H₂S. Same process for cobalt.
538. WÖHLER (C. N., xxii., 84).—From acid solution of German silver precipitate Cu as CuCNS; convert Ni and Zn to double cyanides; precipitate Zn with K₂S (not (NH₄)₂S), and Ni from oxidised filtrate with KHO.
539. MOORE (C. N., lii., 20).—Analysis of German silver. Precipitate Cu from HCl solution with H₂S, add KCN to filtrate, and precipitate Zn with (NH₄)₂S. The Cu and Zn are electro-deposited.
540. *Copper*.—DEWILDE (C. N., vii., 49).—H₂S always carries Ni down. Add cream of tartar, precipitate with KHO in alcohol, re-dissolve in excess, and precipitate Cu with glucose. The Ni, precipitated by KHO, after calcining and grinding, is easily washed free from potash salts.
541. RICKE (C. N., lxxvi., 241).—Ni-Cu alloys (coins) have the Cu deposited from acid solution of sulphates. Then Ni deposited from ammoniacal solution. Similar processes, ROBERTS (J. C. S., i., 101) and LANGBEIN (J. C. S., i., 1077).
542. GLASSEN (J. C. S., xlviii., 191).—Cu electro deposited from ammonio-oxalates.
543. KAIKOW (J. C. S., lxxviii., ii., 245).—Cu precipitated with hydrazine hydrochloride and KI. Ni, Cd, and Zn are in solution. (See also 578, 583).

544. *Arsenic*.—WÖHLER (C. N., xxiv., 141).—Both metals precipitated with Na_2CO_3 , digested with oxalic acid, and Ni or Co oxalates filtered off.
545. JANNASCH and LEHNERT (J. C. S., lxx., ii., 547).—Co and Ni are precipitated from alkaline solution with H_2O_2 .
546. *Cadmium*.—SMITH and WALLACE (J. C. S., lxii., 920).—Cd electro-deposited after adding KHO. Also RUDORFF (J. C. S., lxiv., 305) and SMITH and MOYER (J. C. S., lxiv., ii., 496). Ni is precipitated in acid solution if a strong current is employed.
547. *Bismuth*.—SMITH and MOYER (J. C. S., lxiv., ii., 497).—Electrolytic. H_2SO_4 preferable to HNO_3 solutions.
548. JANNASCH and ROSE (J. C. S., lxvi., ii., 482).—Separation in ammoniacal solution with H_2O_2 .
549. *Mercury*.—SMITH and FRANKEL (J. C. S., lviii., 664).—Electro-separation of Hg and Ag from Ni and Co. Also HEIDENREICH (J. S. C. I., 1896, 774) and RUDORFF (J. C. S., lxvi., ii., 399).
550. *Gold*.—SMITH and MUHR (J. C. S., lx., 1398).—Electro-separation from cyanide solutions.
551. *Aluminium*.—GONTIERRE (J. S. C. I., 1896, 830).—The metallic Al is dissolved in NaHO. The Ni is separated from the residual metals (Cu, Pb, Fe) by electrolysis and precipitation with ammonia.
552. LEFFLER (C. N., lxxvii., 265).—Al precipitated with soda phosphate in acetic acid solutions.
553. *Lead*.—JANNASCH and LESINSKY (J. C. S., lxvi., ii., 33).—Precipitated from ammoniacal solution with H_2O_2 .

III. GRAVIMETRIC ESTIMATION OF NICKEL.

III. 1. As Oxide or Sulphide.—

554. HADOW (C. N., ii., 85).—Ni separated from solution of the ore with CaCO_3 and acetate; Ni and Co precipitated with H_2S and converted into sulphates, or precipitated with KHO or binoxalate and weighed as oxides. Filter-paper absorbs Ni and Co from strong solutions of the acetates.
555. GIBBS (C. N., xvii., 172).—Ni is completely precipitated by a small excess of Na_2CO_3 and boiling. Ignited to oxide.
556. SCHMIDT (C. N., lxx., 248).—Precipitated by H_2S from an $(\text{NH}_4)\text{NO}_3$ solution. The sulphide converted to oxide by ignition with ammoniacal mercuric cyanide.
557. ROSE (C. N., ii., 302).—Neither Ni nor Co can be estimated as sulphide by heating with S in H, as iron, Mn, Zn, and Cu may.
558. FRESSENIUS (C. N., iv., 150).— $(\text{NH}_4)_2\text{S}$ precipitates Ni slowly and not completely; $(\text{NH}_4)\text{Cl}$ assists, $(\text{NH}_4)\text{HO}$ retards, the precipitation.
559. LECREQUIER (J. C. S., lviii., 297).—The retention of nickel sulphide in solution is due to the formation of a thionickelate.
560. TERREIL (J. C. S., lxii., 1132).— H_2S precipitates Ni and Co in the presence of many organic salts and sulphur and phosphorus acids.
561. BAUBIGNY (C. N., xlv., 174 and 229).—Precipitation of NiO salts with H_2S depends on the ratio weight of acid to weight of metal, and not weight of acid to weight of water. This distinction serves to separate other metals (e.g., Zn). (See also C. N., xlv., 257). For action of H_2S on NiSO_4 in acetic solutions see C. N., xlv., 17; for action on NiCl_2 , see C. N., xlv., 40; action on Co salts, J. C. S., liv., 113.

III. 2. By Electro-deposition.—

562. MERRICK (C. N., xxiv., 100, 172).—Cu and Ni may be successively deposited from the same solution. Only a limited number of salts are suitable for electro-deposition (C. N., xxvi., 209).
563. FRESSENIUS and BERGMANN (C. N., xlii., 75).—Co and Ni not deposited if solution contains free mineral acids. Solutions of $(\text{NH}_4)_2\text{SO}_4$ (no NH_4Cl) are

best. Sulphocarbonate used to indicate when precipitation is complete.

564. RUDORFF (J. C. S., lxiv., ii., 94).—From sulphate or pyrophosphate solutions.
565. CLASSEN (J. C. S., xlviii., 191).—Ni, Co, and Fe deposited from double oxalates. Fe estimated volumetrically, Ni by difference; Co separated with nitrite.
566. BRAND (J. C. S., lviii., 294).—From ammoniacal solution of the pyrophosphates.
567. KOHN and WOODGATE (J. S. C. I., 1889, 256).—From various solutions. Table of metals which can be determined quantitatively, and references.
568. CLASSEN (J. C. S., lxvi., ii., 481).—Particulars of current density, potential, &c., for deposition of Ni and other elements.
569. WOLMAN (J. C. S., lxxiv., ii., 50).—Oxalate (565) and pyrophosphate (564) solutions lead to high results.
570. LANGBEIN (C. N., lvii., 251).—The deposit from solutions containing Mn are contaminated thereby.
571. RICHE (C. N., xxxiv., 96).—Deposition from ammoniacal solution carries down Mg. Purified by re-deposition.
572. ETTTEL (J. S. C. I., 1895, 69).—From ammoniacal solution of chlorides. (See also 501, 502, 504, 510, 520, 521).

III. 3. Miscellaneous Processes.—

573. FRESSENIUS (C. N., xxix., 193).—Ore, &c., decomposed by acid or bisulphate, Schwarzenberg separation of Fe. Ni and Co precipitated with H_2S , with KHO, and finally reduced to metal in hydrogen. (See also C. N., xxx., 16). Zn separated by volatilising with $(\text{NH}_4)\text{Cl}$, and Co as nitrate.
574. BAYLEY (J. S. C. I., 1887, 449).—Add $(\text{NH}_4)_2\text{S}$ to alkalinity, ammonium benzoate, and HCl; the precipitated sulphide weighed as sulphate; Zn separated by a solution containing phosphoric acid.
575. CLARKE (J. S. C. I., 1896, 866).—To solution containing ammonium phosphate add HCl to neutrality, then 20 per cent alcohol. Precipitate ignited to pyrophosphate. Zn is estimated similarly. (See also 632).

IV. VOLUMETRIC ESTIMATION OF NICKEL.

IV. 1. Titration with Potassium Cyanide.—

576. LECREQUIER (C. N., lxxi., 188).—Feebly alkaline solution titrated with KCN until the precipitate first formed just re-dissolves; NH_3 salts or small amounts of $(\text{NH}_4)\text{HO}$ do not interfere.
577. MOORE (C. N., lix., 160).—Titrate in ammoniacal solution, using cupric ferrocyanide indicator. Sulphates, nitrates, chlorides, and acetates do not interfere.
578. CAMPBELL (C. N., lxix., 139).—Fe separated as phosphate in acetic solution. Separate Cu with Pb, and then Pb and Mn as phosphate. Deposit Ni electrolytically or titrate with KCN, as in 577. How to prepare the indicator.
579. MOORE (C. N., lix., 292).—Mn and large amounts of ammonium salts interfere with (577). By adding pyrophosphate to acid solution until precipitate re-dissolves and titrating ammoniacal solution, neither Fe, Al, Zn, nor Mn interfere.
580. DENIGES (C. N., lxix., 42).—General method for estimation of Ag, in which KI is added to form indicator in ammoniacal solution. (This very suggestive paper should be read along with the processes for estimating Ni based on the same principle).
581. MOORE (C. N., lxxii., 92).—KCN added until clearing of the suspended AgI indicates the formation of $\text{Ni}(\text{CN})_4$. The KCN may be standardised with Ni or Ag; Co is estimated with the Ni; Mn, Cu, and Zn interfere; Zn, Al, Fe, or Mg kept in

solution with pyrophosphate or an organic acid.* (A good account of an admirable process).

582. BREARLEY and JERVIS (C. N., lxxviii., 77, &c.).—Some points in the cyanometric estimation. Interferences exerted by a score elements, and means of avoiding them. Estimations in presence of elements giving precipitates in alkaline solutions. (See also 495, 511).

IV. 2. Miscellaneous Volumetric Processes.—

583. KUNZELL (C. N., viii., 38).—Hot ammoniacal solution titrated with soda sulphide, using silver or nitro-prusside as indicator. Titration may be made in presence of Cu.
584. SYSSOYEFF (Z. I. S. I., 1893, i., 414).—Precipitate Ni_2O_3 with Cl from a KCN solution; the Ni is calculated from the reaction—



after measuring the oxygen. Iron kept in solution as ferrocyanide.

585. GIORIS (Z. C. S., lxxv., 452).—Gibbs's process, precipitating with alcoholic oxalic solution, is inaccurate. Precipitate with oxalic solution of Sr or Ba oxalate, and titrate either the dissolved precipitate or the excess of precipitant with permanganate.
586. LUCAS (C. N., lxxx., 38).—Separate Fe with ammonia and $(\text{NH}_4)\text{Cl}$, add K sulphocarbonate, and compare red colour formed with standards; Co can be detected by using ammonium sulphocarbonate at the same time.
587. SPULLER (Z. I. S. I., 1897, ii., 507).—Fe precipitated with ZnO ; the filtrate colorimetrically compared with standards.

V. DETECTION OF NICKEL.

588. ALLEN (C. N., xxiii., 290).—Add $(\text{NH}_4)\text{Cl}$, $(\text{NH}_4)\text{HO}$, and ferricyanide; a red colour = Co; no change but a red precipitate on boiling = Ni. (See also CARTER, C. N., xlvii., 273).
589. DAVIES (C. N., xxxii., 44).—After detecting Co with ferricyanide, add ferrocyanide and HCl; a precipitate = Ni; detects 0.01 per cent; ferrocyanide may be used to detect Co also.
590. DONATH and MAYRHOFER (C. N., xlv., 46).—Precipitate with NaHO and I, dissolve out Ni with ammoniacal $(\text{NH}_4)\text{Cl}$, and detect with $(\text{NH}_4)_2\text{S}$. Boil HCl solution of the residue with KHO; blue liquid = Co.
591. VILLIERS (C. N., lxxi., 45 and 49).—Tartaric acid, NaHO, and H_2S added successively. The colour of the filtrate indicates traces of Ni.
592. CAVALLI (Z. C. S., lxxii., 603).—Precipitate with nitroprusside and treat with $(\text{NH}_4)\text{HO}$; only the Ni compound dissolves.

VI. MISCELLANEOUS NOTES.

593. PATTINSON (C. N., viii., 136).—Ni and Co exist in small amounts in Cleveland ironstone.
594. WARREN (C. N., lv., 37).—Certain metallic adulterations of cube Ni render the metal non-magnetic, and may thus be detected.
595. FLITTMANN (C. N., lxx., 280).—Commercially pure nickel contains more or less Fe, Cu, Co, Zn, and Mn. Instructions for quantitative estimation of these.
596. BETTEL (C. N., lviii., 48).—Analysis of nickel-silver and separation of Sn, Pb, Cu, Zn, Fe, and Mn. Ni electro-deposited.

* On this principle nickel is sometimes estimated in the Siemens steel bath, in about ten minutes, in order to find out how much of the rich ferro-nickel alloy must be added to bring the melt (which already contains nickel, due to scrap) up to the desired percentage. The KCN is standardised under the same conditions. Chromium interferes with the end-reaction.

597. CARNOT and GOUTAL (Z. C. S., lxxii., ii., 555).—Nickel is probably simply mixed with, or dissolved in, the iron.

(To be continued).

THE COMBUSTION OF ACETYLENE IN AIR ENRICHED WITH OXYGEN.

By G. L. BOURGEREL.

DURING the course of various researches carried out in the laboratory of the "Société Volta," at Geneva, I had occasion to seek for a source of high temperature other than that of the electric furnace, so that all electro-chemical action might be avoided in the reactions I had occasion to study. As the "Société Volta" were manufacturers of carbide of calcium, I naturally made use of acetylene, which is, further, in general use in the laboratory, and I made use of the burner of a table blowpipe, such as is commonly used for glass-blowing. By using compressed air, or air from a bellows, and acetylene, I produced a very high temperature, by means of which pure nickel or pure gold could be melted with ease. But as this temperature still did not satisfy my wants, I replaced the compressed air by pure compressed oxygen, which can now be easily obtained in cylinders.

Great, however, was my surprise to see that the flame leaving the blowpipe was exceedingly brilliant, and that, as a matter of fact, the gases did not mix, but burned only on contact one with the other, as in a lamp flame, forming a corona of flame, empty in the centre; then little by little there formed at the extremity of the central tube of the blowpipe a deposit of carbon which rapidly increased, taking the form of a truncated cone with the base outwards. This hollow cone, resembling a pear in shape, is formed of very dense carbon, very sonorous and hard at the base of the flame—that is to say, the point of the cone. It is so brilliant that at this point the outside appeared to be almost white, while all the wider portion and the interior has the appearance of very fine, light carbon, similar to lamp-black. Some individual cones were so hard at the narrow part that glass was easily scratched when fragments were rubbed on the surface with the aid of a cork.

The carbon thus deposited, when examined with a lens, has the appearance of an enormous number of cauliflowers crowded together. The inner side of this hollow pear is covered with a thin layer of lamp-black, and under this coating the carbon is perfectly bright and polished, hard, and of a steel-grey colour.

Its fracture is bright steel-grey at the lower portion, and becomes darker towards the large end; it is built up of superimposed layers, separated by very thin films of black, easily visible in the middle parts.

The very brilliant flame produced at the upper end of the cone is also very hot; on placing a piece of carbide of calcium the size of an egg in this flame it becomes red-hot, and fuses, while at the same time swelling up and throwing off on all sides incandescent particles, which continue to burn in the same manner as iron burns in oxygen; this makes a very effective experiment. The temperature of the acetylene which burns in the air round the end of this carbon cone, though not immediately in contact with it, is not very high; it is only at a nascent red-heat, and the examination of this flame quite justifies the theory of the dissociation of the acetylene at the moment of its combustion.

To return to the description of this curious experiment; as I did not want an incomplete combustion in my blowpipe, the idea occurred to me to return to the use of compressed air, and to introduce pure oxygen to enrich the air, by means of a branch tube joining the one which supplied the air.

In this manner the proportions of air and oxygen to each other and to the acetylene could be varied, and

flames of complete combustion produced having a neutral character, so to speak, neither oxidising or reducing; while, on the other hand, by increasing the amount of oxygen very hot oxidising flames could be obtained.

Thus in a non-luminous flame of this latter kind, a morsel of carbide of calcium became dissociated, losing its carbon, and becoming covered with a layer of fused carbonate of lime, a remarkable fact, while a small portion of the calcium is volatilised, and colours the flame red.

By using the blowpipe with a mixture of air and oxygen as I have described, we have at once a convenient source of intense heat, comparable with that of the electric arc, by means of which platinum itself can be melted in a few seconds, and which is free from the drawbacks inherent to the use of the electric arc, which is always reducing and carburising, and to the use of the oxyhydrogen blowpipe; both in commercial work as well as in the laboratory there are immense advantages to be gained by the use of such a source of heat.—*Moniteur Scientifique*, Oct., 1900.

ON OXYGEN IN STEEL.

By L. ROMANOFF.

ALTHOUGH the presence of very small quantities of oxygen has a very great effect on the resistance of steel to rolling, the estimation of this element is very rarely undertaken in metallurgical laboratories. It is important to determine the cause of the difficulties offered by this estimation, which takes a considerable time, and which requires the use of complicated apparatus.

Having had occasion to examine a certain number of samples of steel, burnt during the process of making in the Martin furnace, I decided to estimate the oxygen they contained. As a general rule, the quantity of air admitted to the hearth of the furnace is 10 per cent more than the theoretical amount, with the object of effecting complete combustion with the gases. In the Vacelet furnaces used by us, the gases ought to be consumed much more quickly, since the fuel (naphtha residues) is introduced direct, without any previous preparation. We can only achieve this by admitting as much as 25 per cent too much air into the furnace. At the commencement this method of working gave us burnt steels.

The method now in use of estimating the oxygen in steel was first due to Prof. Ledebur (*Stahl und Eisen*, 1882, p. 193). He made this estimation in various kinds of iron and steel, and determined the influence it exerted on the physical properties of the metal. Unhappily, since his time the progress made in this direction has been practically nil.

Two or three papers by Ledebur (*Stahl und Eisen*, 1883, p. 502; *Chem. Zeit.*, 1885, p. 17). A paper by Gladky (*Stahl und Eisen*, 1893, p. 293), and a few other odd papers (B. C. Calvert, *Comptes Rendus*, lxx., p. 453; Akerman, *Stahl und Eisen*, 1883, p. 149; Stummer, *Pogg. Anal.*, lxxii., p. 136; Gruner, *Ann. Chim. Phys.*, Series 4, xxvii., p. 5; Platz, *Stahl und Eisen*, 1885, p. 471; Gladky, *Monit. Scient.*, 1889, p. 755) are the only literature we possess on this important question.

Before describing the methods we now have at our disposition for estimating the oxygen in steel, I must mention that all of them possess sources of error, and also entail very complicated manipulation. These methods may be divided into three groups:—

The first method depends on the property possessed by dry chlorine of acting on iron without touching its oxide. A current of dry chlorine is therefore directed on to some steel turnings or filings, so as to remove the iron in the form of chloride; unfortunately the residue does not consist entirely of oxide of iron—it also contains carbon, oxide of manganese, and a series of other compounds not attacked by chlorine.

The second method is founded on the use of a dissolving agent such as the sulphate or chloride of copper, iodine, bromine, bichloride of mercury, &c. Here again, however, the residue is not composed solely of pure oxide of iron; it also contains carbon, oxide of manganese, phosphorised and sulphurised compounds of iron, &c.

The third method, devised by Ledebur, consists of passing a current of pure dry hydrogen over steel turnings at a red heat, and weighing the water formed. It is, of course, necessary that the sample submitted to this treatment should be first made perfectly clean. If the metal contains a little oxide of manganese, that at once is a cause of error. The estimation of the oxide of iron in steel is of very great interest, since, according to Ledebur, it is this substance alone which possesses a deleterious action (Ledebur, *Moniteur Scientifique*, 1885, p. 732). In fact, according to this author, oxide of manganese occurs in melted steel solely in the form of a mechanical mixture, while the oxide of iron is in the dissolved state. By combining the third method with either of the two preceding ones we can obtain exact results; but such a proceeding would be extremely complicated, and could not well be carried out practically.

I must say a few words about another method, due to Tucker (*Iron and Steel Institute*, 1881, p. 205). It consists of mixing steel filings with powdered charcoal, and calcining the whole in a crucible. The steel is weighed before and after calcination, and the loss of weight represents the oxygen. Besides the fact that by this method we are obliged to use a very large sample (about 1 kilogramme), we are unable to take any account of the gases dissolved in the steel. This circumstance alone may falsify the results to the extent of 50 or even 100 per cent.

After having tried all the above methods, I came to the conclusion that Ledebur's (heating in a current of hydrogen) is the best; I, however, never first heated my samples in nitrogen; besides complicating the analysis without apparently affecting the results, I would point out that it is extremely difficult to prepare nitrogen absolutely free from oxygen. According to Ledebur the preliminary heating in nitrogen is to drive off the hydrocarbides with which the samples are always tainted, but without doing this I have obtained practically the same results as he did. Instead, I always wash the sample in alcohol and ether, and dry it in an exsiccator. In some special experiments, to which I shall refer later on, I did, however, have recourse to heating in nitrogen. As for the presence of hydrocarbides in the steel, during the heating in hydrogen, I do not attribute them to the oil present on the tool or file, but to a totally different cause, about which I shall have more to say further on.

I need not give detailed descriptions of the methods of drying and purifying the hydrogen and nitrogen. I will only say that I followed Ledebur's instructions closely. To the results obtained, and which are given in the following table, I have added a few observations on the behaviour of the metal while being filed. These remarks, though perhaps a little vague, are nevertheless worthy of attention.

I also thought it advisable to give the density of the different steels submitted to analysis, besides the estimation of the oxygen. I must here point out that the density is not so much influenced by the absolute quantity of foreign metalloids (especially oxygen) present as by the state in which this oxygen occurs in the metal, especially in the case of a Siemens-Martin or similar steel. In such a case, the bath of steel, which contains both carbon and oxygen at a very high temperature, cannot remain in a state of repose; the carbonic oxide is formed proportionally to the temperature and to the quantity of oxygen present in the charge. On cooling, the steel is unable to give off all this carbonic oxide, and there always remains therefore a certain quantity mechanically mixed: this will explain the slight variations which occur in the density of the metal.

In determining the densities I used crushed steel, which had passed respectively through two sieves of 1 m.m. and 2 m.m., so as to obtain grains of a uniform size. The grains were carefully stirred in water, after which the air was drawn off by means of a pump. I always used steel which had not been filed; this has the advantage of having a structure as uniform as possible.

No.	Per cent.					Density.	Effect of filing.	Elec. resistance.
	C.	Ph.	Mn.	S.	O.			
—	0.08	0.07	0.20	0.08	0.29	7.69	Crushed	—
496	0.107	0.04	0.57	0.03	0.19	7.79	Much heated	—
498 a	0.25	0.01	0.54	0.03	0.15	7.85	—	11.09
572 a	0.25	0.02	0.55	0.04	0.29	7.72	—	11.28
516.	0.10	0.04	0.50	0.07	0.11	7.92	Good	—
503.	0.10	0.08	0.55	0.06	0.25	7.84	Much heated	—
526.	0.095	0.02	0.37	0.06	0.22	7.78	"	—
540.	0.10	0.04	0.38	0.07	0.16	7.82	Flakey	—
547.	0.095	0.04	0.49	0.05	0.11	—	Good	—
550.	0.105	0.05	0.49	0.03	0.14	—	Rather heated	—
551.	0.09	0.06	0.32	0.04	0.16	7.87	"	—
553.	0.095	0.08	0.39	0.04	0.13	7.80	"	—
555.	0.10	0.05	0.44	0.03	0.13	7.87	"	—
570.	0.095	0.02	—	—	0.03	8.08	Good	—
581.	0.105	0.06	0.46	—	0.09	7.97	"	—
583.	0.10	0.04	0.42	0.03	0.02	8.08	"	—
616.	0.085	0.01	0.37	—	0.00	—	"	—
654.	0.11	—	—	—	0.00	—	"	—
576.	0.095	0.06	0.50	—	—	7.76	Flakey	—
750.	0.09	0.035	0.24	0.05	—	7.77	Crushed	—
— b	0.43	0.02	0.61	0.03	0.07	—	"	—
— c	—	—	—	—	0.05	—	"	—

From the above results we may draw the following conclusions:—

I. The proportion of oxygen varies from 0 to 0.29 per cent; this latter figure appears to be a maximum—we may therefore agree with Ledebur's observation that 0.29 per cent of oxygen corresponds to the limit of solubility of the suboxide of iron (1.30 per cent) in steel.

II. A steel, to be easily filed, should not contain more than 0.10 per cent of oxygen.

III. The proportion of oxygen present has a distinct connection with the variation in the density.

Thus we see that an easily fileable steel, containing not more than 0.11 per cent of oxygen, has a minimum density of 7.90. A steel containing more than 0.20 per cent of oxygen, and having a density below 7.80, must be looked upon as behaving very badly under the file.

It is curious to note that the samples Nos. 498 and 572, intended for telegraph wires, gave such different results from the point of view of electrical resistance. Sample No. 498, containing 0.15 per cent of oxygen, and having a density of 7.85, had an electric resistance of 11.09 ohms on a wire of 4 m.m. diameter, which fulfils the requirements of the telegraphic department. On the other hand, No. 572, containing 0.29 per cent of oxygen, with a density of 7.72, had a resistance of 11.28 ohms, or 0.19 above the standard. The cause of this difference is to be found in the lower density of the metal, which itself results from the high proportion of oxygen present.

For the closer study of the important part played by oxygen in steel, I undertook a series of estimations of oxygen on the same sample of steel during different phases of its refining. I hoped thus to decide why any given type of steel contained more oxygen than others. At the same time I was enabled to determine the exact part played by ferro-manganese in the refining of steel.

For this purpose I chose sample No. 616. It was made from 2457 kilos. of scrap-steel and 819 kilos. of cast-steel from the Novorossiysk Co. These materials were taken on account of the low proportions of silicon, sulphur, and phosphorus they contained, so that the influence of these elements would not interfere with the experiments in question.

A sample was taken every ten minutes. The two first

were taken from the cold metal, the others at different stages of the refining.

The first addition of ferro-manganese, made after 6 hours 45 minutes, consisted of 12.3 kilos.; the second, after 7 hours 5 minutes, consisted of 24.6 kilos. The original charge was completely melted 5 hours 50 minutes after taking the first sample. The metal was run after 7 hours 15 minutes.

The results of these assays are given in the following table. I have already pointed out that the loss of weight undergone by the filings heated in hydrogen is greater than the weight of water collected, and I attribute this difference to the evolution of the gas dissolved in the metal. I have therefore tabulated this item as well as the proportion of oxygen present.

Sample No. 616.

Time of taking the sample.		Oxygen. Per cent.	Gas dissolved. Per cent.	Remarks.
Hrs.	Mins.			
5	50	0.14	0.52	Incompletely melted.
6	00	0.19	0.39	
6	10	0.39	0.65	Completely melted.
6	20	0.34	0.56	
6	30	0.27	0.45	
6	40	0.21	0.35	
6	45	First addition of ferro-manganese.		
6	50	0.05	0.43	
7	0	0.02	0.22	
7	5	Second addition of ferro-manganese.		
7	10	0.00	0.33	
7	20	—	0.11	

In the examination of the results of the estimation of the oxygen, we must first of all state that the two first samples taken cannot be considered as representative of the average composition of the charge. In fact, at the time when these samples were taken, a part of the mass, containing suboxide of iron, still remained on the floor of the furnace unmelted. The sample consisted chiefly of the cast-steel, hence its low proportion of oxygen. We see, further, that when the mass is completely melted (6 hours 10 minutes) the proportion of oxygen immediately becomes much higher (0.39 per cent instead of 0.14 per cent). These two points have been confirmed, not only by analysis, but also by observations made on the state of the charge at different times during the operation.

As soon as the oxygen has obtained its maximum (0.39 per cent), which may be taken as its normal value, its proportion decreases rapidly at the same time as the oxide of iron itself, and that at the expense of the carbon. This gradual diminution continues until the first addition of ferro-manganese. As was to be expected, this addition has the effect of still further deoxidising the bath, and the second addition of ferro-manganese reduces the oxygen present to zero.

The action of the ferro-manganese is well known. The manganese, being more easily oxidised than the iron, takes up oxygen at the expense of the suboxide of iron in the bath, and passes in its turn into the state of oxide which is found in the slag.

If the operation is prolonged after the disappearance of the greater part of the manganese, the proportion of oxygen will be seen to rise again, since the deoxidising elements—manganese and carbon—would be completely eliminated. In this way we can explain the fact that the softer the steel is, and the less reducing agents it contains (manganese, phosphorus, silicon), the more easily it is burnt. If the ferro-manganese is added too soon, or in too small quantity, it passes into the slag, and the iron—no longer being protected against oxidation—ends by containing a considerable quantity of oxygen.

As for the proportion of dissolved gas, we can see, from the above table, that it diminishes at the same time as the oxygen. At the moment of adding the ferro-manganese it rises rapidly, to again fall in a regular manner. This result was expected; in fact, by the passing of the oxides

into the slag the ferro-manganese encourages the evolution of carbonic oxide, which thus increases the proportion of gas dissolved.

We must also draw attention to the rapid fall in oxygen (0.33 to 0.11 per cent) during the time following the running of the metal. On arriving in the mould the metal contains a high proportion of gas, which is dissolved at a higher temperature, and of which the solubility is less in the cold. This gas is therefore given off rapidly as the temperature of the metal falls. It follows that it is preferable to wait till the boiling which follows the addition of the ferro-manganese is finished, if we wish the casting to be properly made.

To return to the question of the estimation of the oxygen, I have a few remarks to make on the method I was forced to use by reason of not having a better one at my disposal. First we see that the weight of water collected does not correspond exactly with the weight of oxygen contained in the steel. The second figure (gas dissolved) is always higher than the first (oxygen contained).

Further practice in this kind of analysis has shown me that the sulphur and phosphorus contained in the steel form volatile compounds with the hydrogen. This characteristic has already been pointed out by Thidier (*Gorny Journal*, 1883, vi., 458), who worked with moist hydrogen. The phenomenon remains the same, though much less noticeable, with dry hydrogen. Under these conditions it is impossible to control the results obtained by weighing the water.

I now return to a question I have already mentioned. According to Ledebur, carbides of hydrogen are formed when a steel-turning is heated in a current of hydrogen. This author maintains that the formation of these carbides is accounted for by the presence of oil or grease in the steel, coming from the tool used for cutting it (*Stahl und Eisen*, 1882, p. 194).

To verify this point I operated on the three following samples:—

1. Soft steel containing 0.15 per cent of carbon.
2. Hard steel containing 0.43 per cent of carbon.
3. Cast-steel containing 0.48 per cent of combined carbon, 2.90 per cent of graphite.

These three samples were heated for half-an-hour, at a red heat, in a current of pure nitrogen. After cooling in nitrogen, the samples were washed in alcohol and ether, and dried. Calcination then gave the following results:—

1. Soft steel.—0.18 per cent of volatile hydrocarbons; 0.09 per cent of non-volatile hydrocarbons.
2. Hard steel.—0.05 per cent of volatile hydrocarbons; 0.02 per cent of non-volatile hydrocarbons.
3. Cast-steel.—0.05 per cent of volatile hydrocarbons; 0.02 per cent of non-volatile hydrocarbons.

I then heated these same samples, for the same length of time, in a current of hydrogen, and hydrocarbides were still formed; from this we must conclude that these compounds are due to the action of the hydrogen on the carbon in the metal. The following are the results obtained by analysis of the samples:—

For the soft steel, the proportion of carbon fell from 0.15 to 0.11 per cent after heating in hydrogen. For the hard steel, it fell from 0.43 to 0.37 per cent. For the cast-steel the combined carbon had not changed, but the proportion of graphite fell from 2.90 to 2.35 per cent.

These variations are still more marked if we prolong the heating in hydrogen. At the commencement the hydrocarbons form fairly quickly; this action then gradually becomes slower, but it cannot be said that it entirely ceases so long as the metal still retains any carbon. When we remember that carbon forms more or less stable compounds with iron, it is not astonishing that hydrogen, in its turn, forms compounds with carbon more or less rapidly. Of these compounds some are volatile and others non-volatile. In the method proposed by Ledebur the non-volatile hydrocarbides are weighed at the same time as the water; the result, as oxygen, is therefore always too high.

A more exact and rapid method is much to be desired; it would enable us to estimate the oxygen in steel with greater accuracy, and to elucidate in a more definite manner the part played by this element in metallurgical products.—*Stahl und Eisen*, 1899, p. 265.

THE ACTION OF WOOD-CHARCOAL ON THE ORGANIC MATTER IN WATER.

By F. MALMÉJAC.

THREE series of experiments have been carried out.

For the first series we took, by the aid of a pair of pincers, several pieces of wood-charcoal from the centre of a large piece, and carefully blew off all the dust. 20 grms. per litre of this charcoal were placed in water, of which the proportion of organic matter present was known.

For the second series, similar pieces of charcoal from the same lump were carefully washed with boiling distilled water, then placed in the same proportion in a like amount of the same water.

For the third series, the same quantity of charcoal was placed in another equal quantity of the same water, after having been first heated to bright redness.

After a preliminary shaking the water becomes permeated with a very light carbonaceous dust, which requires a very long time to settle, and which should be filtered off before making the estimation of the organic matter. The carbon, heated to redness, also introduces small quantities of ash into the water; this gives the water a milky appearance. Before estimating the organic matter, the suspended particles should be removed by filtering through ordinary filter-paper, previously washed with boiling distilled water.

After four hours' contact, during which the waters were shaken a number of times for an equal period, the organic matter was estimated by Levy's method, and the following results were obtained as a mean of each series of experiments:—

	Organic matter.		Time of contact.
	Alkaline solution.	Acid solution.	
Check sample	3.4	4.6	4
Water treated with unwashed charcoal.. .. .	3.4	5.0	4
Water treated with washed charcoal.. .. .	2.0	3.8	4
Water treated with charcoal heated to redness.. .. .	1.8	3.2	4

The above results are expressed in m.grms. per litre.

1. After four hours the purification by means of unwashed charcoal has not yet commenced; further, this carbon has even introduced a small amount of organic matter into the water, determinable by permanganate in acid solution. This drawback is one that can rarely be avoided when using raw charcoal for the purification of water intended for immediate drinking purposes.

2. The action of the charcoal, washed with boiling distilled water, is shown by a very distinct diminution of the organic matter in the water.

3. The same is the case with the charcoal heated to redness. The purifying power of this last is superior to that of the washed charcoal; after four hours' contact it removes about 50 per cent of the determinable organic matter in alkaline solution, and 33 per cent of that determinable in acid solution.

From these results we may conclude that by the last two methods (washed charcoal, and charcoal heated to redness), we can effect a purification which, with moderately contaminated waters, would give sufficiently good results in a few hours, as far as the organic matter goes. It is advisable not to make use of pieces of charcoal simply freed from surface dust if the water is to be used shortly after treatment.

The action of the charcoal, however, does not stop there; its purifying power continues for a considerable time longer; thus, unwashed charcoal which has no effect after four hours contact, gave on succeeding days a more and more pronounced purification, as shown by the following table:—

Organic matter in m.grms. per litre of water after purification for—

Four hours..	Alkali	3.4
	Acid	5.0
Twenty-four hours	Alkali	2.6
	Acid	3.0
Five days	Alkali	2.0
	Acid	2.2
Eight days	Alkali	2.0
	Acid	2.2
Ten days	Alkali	2.0
	Acid	2.2

These results show that the action of charcoal does not continue indefinitely; according to our experiments its action appears to be limited to five days. We might ask whether there do not exist in the water organic matters non-absorbable by charcoal.

To test this hypothesis, we separated the charcoal which had no further action from the purified water, by passing it through a washed filter, and we added to the water thus obtained a fresh quantity of 20 grms. of charcoal per litre. After five days' contact, with occasional shaking, the organic matter in this water was estimated, and the following results were obtained as the mean of the series:—

Organic matter in m.grms. per litre of water	Alkaline	1.2
	Acid	1.0

After further treatment with charcoal for five days we obtained:—

Organic matter in m.grms. per litre of water	Alkaline	0.8
	Acid	0.6

Finally, a third treatment with charcoal for five days gave the same results.

These experiments show that:—

1. Organic matter, non-absorbable by charcoal, may exist in water.

2. 60 grms. of charcoal, employed successively in 20 grm. doses, does not give three times the purification of the first 20 grms.

It remained to find out whether a double dose of charcoal would give twice as good a result from the point of view of the purification of the water.

For this purpose we made two parallel series of experiments on the same water, using 20 grms. of charcoal per litre for the first and 40 grms. with the second.

When the quantity of charcoal is doubled the purification is not doubled; it would be even of advantage to use successive doses if that did not increase the time required.

Heavy, knotty charcoal absorbs organic matter less rapidly than light smooth charcoal.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xii.

THE IODOMETRIC ESTIMATION OF SUGAR BY MEANS OF FEHLING'S SOLUTION.

By N. SCHVORL.

AFTER Lehmann and Riegler followed Maquenne, who also published a method for the volumetric estimation of sugar by means of Fehling's solution, but it seems to me, contrary to the opinions of these authors, that it is the best to prepare the Fehling's solution according to the old formula of Soxhlet-Meissl; that is to say, containing 5 per cent of soda, and only to take 20 c.c. for each estimation.

Further, the methods described by the above-mentioned authors being vitiated by several errors of more or less im-

portance, I adopted the following modification:—10 c.c. of solution of sulphate of copper (69.28 grms. to 1000 c.c.; 10 c.c. = 27.74 c.c. $\text{Na}_2\text{S}_2\text{O}_3$), and 10 c.c. of tartrate solution (346 grms. of Seignette salt and 100 grms. of soda to 1000 c.c.), are run into an Erlenmeyer matras of 200 c.c. capacity; 50 c.c. of distilled water are added, and the whole boiled for two minutes. After cooling the solution under the tap, 10 c.c. of a solution of iodide of potassium at 20 per cent are added, followed by 10 c.c. of sulphuric acid at 25 per cent (1½ vols. of concentrated H_2SO_4 to 8½ vols. of H_2O). The iodine liberated is immediately titrated by means of a decinormal solution of hyposulphite of soda.

Having thus determined the value of the Fehling's solution, we proceed in the same manner with the estimation of the sugar, using such a volume of sugar solution that contains at the most 90 m.grms. of inverted glucose or saccharose, or 125 m.grms. of lactose. The difference between the two titrations represents the quantity of sugar sought for.

When estimating lactose the solution should be kept boiling for five minutes, and it is evident that in this case the boiling must be kept up for the same number of minutes in the determination of the strength of the Fehling's solution.

The following table gives the results obtained with lactose, glucose, saccharose, and starch.

C.c. of N/10 $\text{Na}_2\text{S}_2\text{O}_3$	Lactose.	Glucose.	Saccharose.	Starch.
1	4.5	3.2	3.1	2.8
2	9.0	6.3	6.2	5.7
3	13.6	9.4	9.3	8.5
4	18.1	12.6	12.4	11.4
5	22.7	15.9	15.6	14.3
6	27.3	19.2	18.8	17.3
7	31.9	22.4	22.0	22.0
8	36.5	25.6	25.2	23.1
9	41.1	28.9	28.4	26.1
10	45.8	32.3	31.7	29.1
11	50.6	35.7	35.0	32.1
12	55.4	39.0	38.3	35.1
13	60.3	42.4	41.6	38.2
14	65.2	45.8	44.9	41.3
15	70.1	49.3	48.2	44.4
16	75.0	52.8	51.6	47.5
17	80.0	56.3	55.1	50.7
18	85.0	59.8	58.7	53.9
19	90.0	63.3	62.3	57.1
20	95.0	66.9	65.9	60.3
21	100.0	70.7	69.6	63.7
22	105.1	74.5	73.3	67.1
23	110.4	78.5	77.1	70.7
24	105.9	82.6	80.9	74.3
25	121.6	86.6	84.7	77.9
26	127.5	90.7	88.6	81.6
27	133.8	94.8	92.5	85.3

—*Zeitschrift für Angewandte Chemie*, vol. xii., p. 633.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING SEPTEMBER 30TH, 1900.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, October 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 200 samples of water collected by us during the past month, at the several places and on the several days indicated, from the

mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Sept. 1st to Sept. 29th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 200 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during the month has been remarkably low; rain fell on five days only, viz., the 1st, 26th, 27th, 28th, and 30th; the total being 0.41 inch. The average rainfall for September is 2.43 inches; we thus have a deficit of 2.02 inches, increasing the previous deficit for the year to 2.05 inches, or 11.11 per cent on the thirty years' average.

Our bacteriological examinations of 521 samples have given the results recorded in the following table; we have also examined 34 other samples, from special standpipes, &c., making a total of 555 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 25 samples) ..	121
New River, filtered (mean of 120 samples) ..	10
Thames, unfiltered (mean of 25 samples) ..	4332
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 284 samples) ..	14
Ditto ditto highest	180
Ditto ditto lowest	0
River Lea, unfiltered (mean of 25 samples) ..	158
River Lea, from the East London Company's clear-water wells (mean of 42 samples) ..	13

Of the 446 samples of filtered water supplied to London, examined bacteriologically during the past month, only nine samples, or 2 per cent of the whole number of samples examined, contained between 100 and 181 microbes per c.c.

The exceptionally small rainfall in the Thames Valley during the month no doubt has contributed largely to maintain the relatively high standard of purity of the London waters for this season of the year.

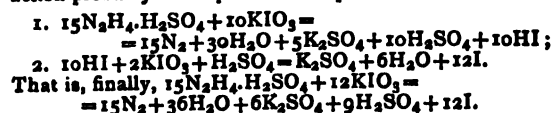
We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.

JAMES DEWAR.

New Method for the Estimation of Hydrazine.—H. Rimini.—If we cause a salt of hydrazine to react on an iodate, an evolution of nitrogen takes place, and at the same time the liquid becomes acid and contains free iodine. The liberated nitrogen or iodine can either of them be estimated, but it is simpler to drive off this latter by boiling, and estimate the remaining iodate. The reaction probably takes place in two phases:—



The bromates behave in the same manner, but only when heated; the chlorates are not decomposed.

The author proposes to extend his examination of this reaction to the estimation of hydroxylamine and semicarbazide.—*Gazz. Chim. Ital.*, (1), vol. xxix., p. 265.

THE SEPARATION AND DETERMINATION OF MERCURY AS MERCURIUS OXALATE.*

By C. A. PETERS.

It is stated in the literature (Rose and Finkener, *Handbuch der Analytischen Chemie*, i., 319) that oxalic acid, neutral and acid oxalates of the alkalis, precipitate mercurous salts, and that oxalic acid and the double oxalates of potassium produce no precipitate with mercuric chloride solution. Starting with these facts, the attempt was made to estimate mercurous salts: volumetrically, by precipitating with ammonium oxalate and determining the oxalic acid by potassium permanganate; and gravimetrically, by direct weighing of the precipitate.

The Volumetric Estimation.

The mercurous nitrate solution used was standardised by the battery, and contained about 12 grms. of metallic mercury to the litre. To obviate the tendency of the mercury salt to break down and form basic salts (Graham Otto, *Handbuch*, iii., 1102) upon the addition of a large amount of water if no nitric acid is added, the solution is prepared in the following manner:—About 20 grms. of mercurous nitrate were ground in a mortar, transferred to a flask, and 200–300 c.m.³ water added. After shaking well, the solution was filtered, and the filtrate diluted to 1 litre. Five c.m.³ of this solution, when precipitated with a sodium chloride solution, gave a filtrate from which only a very slight darkening in colour could be obtained, even upon several hours standing, when treated with hydrogen sulphide, thus showing the absence of a mercuric salt.

A solution made in the above manner had not changed its standard after a period of eight weeks. The potassium permanganate solution (approximately $N/10$) was standardised against lead oxalate.

It was first attempted to estimate the mercurous salts as follows:—The mercurous oxalate was precipitated cold by means of ammonium oxalate, stirred well, and allowed to settle, the completion of the precipitation being determined by addition of more ammonium oxalate. The precipitate was collected on asbestos, washed once or twice with cold water, and (still in the crucible) treated in a beaker with 5 c.m.³ of strong hydrochloric acid. To the solution diluted to 100–200 c.m.³ 1 gram. of a manganous salt was added, and the oxalic acid was titrated with permanganate at the ordinary temperature of the room. The end colour was not stable and was hard to determine. Three experiments, using 0.1217 of mercury in form of mercurous nitrate, gave plus errors of 0.0011 gm., 0.0017 gm., and 0.0028 gm. respectively, or 1.5 per cent. The precipitate when dissolved in sulphuric acid and titrated gave no better results. To obviate this difficulty, the ammonium oxalate solution was matched on the permanganate and the oxalic acid in the filtrate determined. The results obtained by this method, given in the following table, are quite accurate.

Hg taken as Hg(NO ₃) ₂	Hg found.	Excess of ammo- nium oxalate (sp. gr. approx. 1.10).	HCl (1:10).	H ₂ SO ₄ (1:1).	Error as Hg.
Grm.	Grm.	C.m. ³ .	C.m. ³ .	Grm.	C.m. ³ .
A.					
1. 0.1825	0.1823	0.90	5	0.5	— 0.0002
2. 0.1217	0.1218	0.93	5	0.5	+0.0001
3. 0.1217	0.1206	0.99	5	0.5	— 0.0011
4. 0.1217	0.1210	4.05	5	0.5	— 0.0007
5. 0.3042	0.3034	4.97	5	0.5	— 0.0008
B.					
6. 0.1217	0.1220	0.93	—	—	5 +0.0003
7. 0.1217	0.1211	0.97	—	—	5 —0.0006
8. 0.1825	0.1827	0.89	—	—	5 +0.0002
9. 0.3042	0.3040	0.82	—	—	5 —0.0002
10. 0.1217	0.1202	4.10	—	—	5 —0.0015

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, vol. ix., p. 402.

In the experiments recorded in section A of the table the filtrate was titrated in the presence of hydrochloric acid and a manganous salt, at a temperature of 20–40° (Gooch and Peters, *Amer. Journ. Sci.*, 1899, vii., 461). In the experiments under section B, sulphuric acid was added, and the solution heated in the usual manner. An excess of ammonium oxalate, as shown in experiments 4, 5, and 10, interferes in no way.

The separation of mercurous salt from small quantities of mercuric salts, by the means of dilute nitric acid, sp. gr. 1.15, was next attempted. It is stated (Souhay and Lenssen, *Ann. d. Chem.*, cil., 43) that mercurous oxalate is insoluble in cold dilute nitric acid, while mercuric oxalate is more or less soluble in the same reagent. Before attempting any separation, however, there are three factors with reference to the action of the nitric acid which need to be determined: first, the maximum amount of nitric acid that may be present in titration of an oxalate without interference; second, the maximum amount of nitric acid that may be present in a precipitation of mercurous oxalate without having any perceptible solvent action upon the same; and, third, the amount of mercuric oxalate which will be held in solution by given amounts of nitric acid.

To determine the amount of nitric acid that may be present in the titration of an oxalate, 10 c.m.³ of $\frac{n}{10}$ ammonium oxalate were titrated with permanganate at a dilution of 100 c.m.³ with sulphuric acid at 80° C. The event proved that 10 c.m.³ of nitric acid, sp. gr. 1.15, may be present without appearance of interfering action. The maximum amount of nitric acid which may be present without action upon the mercurous salt was determined as shown in the following experiments:—

Hg taken as $Hg_2(NO_3)_2$.	Excess of ammonium oxalate (sp. gr. approximately 1.15).	HNO_3 (sp. gr. 1.15).	Volume precipitation.	Hg found.	Error.
Grm.	C.m. ³ .	C.m. ³ .	Grm.		
0.1122	1.64	10	100	0.1087	–0.0035
0.1122	2.62	6	100	0.1098	–0.0024
0.1122	1.59	6	100	0.1096	–0.0026
0.1122	1.53	5	100	0.1109	–0.0013
0.1122	1.40	5	100	0.1134	+0.0012
0.1122	1.50	5	100	0.1115	–0.0007
0.1122	1.70	4	100	0.1116	–0.0006
0.1122	1.59	8	200	0.1096	–0.0036
0.1122	6.62	8	200	0.1111	–0.0011
0.1122	7.72	8	200	0.1108	–0.0014
0.1122	2.59	5	200	0.1107	–0.0015
0.1010	2.07	5	200	0.1003	–0.0007

Working under the conditions stated in the above table, it is plain that 5 c.m.³ of nitric acid (sp. gr. 1.15) may be used before its solvent action is sufficient to interfere with the accuracy of the process.

To determine the amount of mercuric salt that would be held up by 5 c.m.³ of nitric acid, sp. gr. 1.15, the following experiments were made:—

Hg taken as $Hg_2(NO_3)_2$.	HNO_3 (sp. gr. 1.15).	Ammonium oxalate (sp. gr. 1.15).	Volume.	Time before precipitation.
Grm.	C.m. ³ .	C.m. ³ .	C.m. ³ .	
0.0335	4	0.75	100	5 hours.
0.0095	4	1.00	100	No precipitate 20 hours.
0.0143	5	6.5	100	" "
0.0238	5	1.5	100	Slight precipitate 20 hrs.
0.0238	5	5.5	100	25 minutes.

Five c.m.³ of dilute nitric acid (sp. gr. 1.15) will prevent the precipitation of small amounts of mercuric salt, 10 to 20 m.grms. calculated as mercury, depending upon the amount of ammonium oxalate present in excess. This amount of nitric acid has no apparent solvent action on a precipitate of mercurous oxalate under conditions already

stated, and does not interfere with the titration of an oxalate by permanganate, as already shown.

Carrying out the process of separation of mercurous from mercuric salts, the precipitation was made as described for the estimation of mercurous salts alone, excepting that nitric acid and mercuric salt were added. The experiments in A, B, and C of the accompanying table show the amounts of mercuric salt from which the mercurous oxalate may be separated with 2 c.m.³ of nitric acid. In experiments in Section A the results are quite accurate, but an excess of ammonium oxalate tends to increase the results a little, as seen in experiments under B. An increase in the amount of mercuric salt present causes also, as shown in Section C, the results to be a trifle high.

Hg taken as $Hg_2(NO_3)_2$.	$Hg(NO_3)_2$ present as Hg.	Ammonium oxalate (sp. gr. 1.15).	HNO_3 (sp. gr. 1.15).	Vol. at precipitation.	Hg found.	Error.
Grm.	Grm.	C.m. ³ .	C.m. ³ .		Grm.	
A.						
0.1217	0.0067	0.86	2	100	0.1232	–0.0015
0.1217	0.0067	0.92	2	100	0.1220	+0.0003
0.1217	0.0067	0.97	2	100	0.1218	+0.0001
0.1217	0.0067	0.90	2	100	0.1224	+0.0007
0.1217	0.0067	0.91	2	100	0.1213	–0.0004
B.						
0.1221	0.0067	3.92	2	100	0.1237	+0.0016
0.1217	0.0067	3.93	2	100	0.1235	+0.0018
0.1242	0.0067	8.75	2	100	0.1263	+0.0021
C.						
0.1217	0.0134	0.87	2	100	0.1230	+0.0013
0.1217	0.0134	0.86	2	100	0.1232	+0.0015
D.						
0.1217	0.0134	3.93	4	100	0.1218	+0.0001
0.1217	0.0134	3.90	4	100	0.1211	–0.0006
E.						
0.2244	0.0067	1.88	4	115	0.2244	±0.0000
0.2244	0.0067	1.91	4	130	0.2240	–0.0004
EE.						
0.2244	0.0141	2.98	4	100	0.2230	–0.0014
0.2244	0.0141	2.94	4	100	0.2241	–0.0003
F.						
0.2244	0.0067	8.88	4	100	0.2289	+0.0045
0.2244	0.0067	8.90	4	100	0.2285	+0.0041
G.						
0.2424	0.0067	1.75	4	200	0.2432	+0.0008
0.2424	0.0067	2.00	4	200	0.2414	–0.0010
0.2424	0.0067	2.96	4	200	0.2421	–0.0003
H.						
0.2245	0.0134	1.74	4	200	0.2271	+0.0026
0.2245	0.0134	1.81	4	200	0.2256	+0.0012
I.						
0.1122	0.0144	6.62	5	200	0.1121	–0.0001
K.						
0.1122	0.0240	6.54	5	200	0.1136	+0.0014
0.1122	0.0240	6.57	5	200	0.1130	+0.0008

In experiments D, using 4 c.m.³ of nitric acid even in the presence of an excess of ammonium oxalate, the results are accurate. In experiments E the amount of mercurous salt was doubled and the results are accurate. In EE the amount of mercuric salt was also doubled, and the results are still fairly accurate; but when a large excess of ammonium oxalate is present, as in experiments F, even with the smaller amount of mercuric salt, the results are high. At a dilution of 200 c.m.³ the results are normal as seen in experiments G; but the introduction of more mercuric salt, as in experiment H, causes a plus error.

Using 5 c.m.³ of nitric acid, the larger amount of mercuric salt, together with a large excess of ammonium

oxalate, as recorded in experiments K, the error is raised a trifle; but with a smaller amount of mercurous salt as in experiment I the result is normal.

In precipitating mercurous salts by ammonium oxalate ($\frac{w}{10}$) it is an easy matter to keep the excess of the precipitant within the limits of 1 or 2 c.m.³, because the mercurous oxalate, when properly stirred, settled very rapidly.

The Gravimetric Estimation.

All the conditions described above in the volumetric estimation of mercurous oxalate, for the separation of mercurous from mercuric salts, may be applied to the gravimetric estimation of mercurous oxalate. The precipitate is collected on a weighed asbestos filter, washed two or three times with cold water and dried over sulphuric acid to a constant weight. Amounts of mercurous oxalate equivalent to 0.1217 and 0.2244 grm. of metallic mercury dried to a constant weight over sulphuric acid in about fifteen hours; a larger amount equivalent to 0.3 grm. of metallic mercury, required about two days to dry to a constant weight. Souchay and Lenssen (*Ann.*, ciii., 308), state that mercurous oxalate breaks up at 100°; consequently this temperature cannot be used for drying. For example, a precipitate containing 0.1122 grm. mercury as the oxalate which when brought to a constant weight at the ordinary temperature over sulphuric acid weighed 0.1371 grm., and was heated and weighed as follows:—

After 7 hours at 110°, weight = 0.1338 grm.

" 2 " " " 0.1328 "

" 7 " " " 0.1302 "

The result shows a loss of 0.0069 grm. for sixteen hours heating, and agrees with the statement of Souchay and Lenssen.

The following experiments give the results of the gravimetric work, in which the drying was effected, by exposure for fifteen hours or less, at ordinary temperatures, over sulphuric acid. The larger amounts of nitric acid present in the separations cause the precipitate to be more granular and aid in the filtering process:—

Hg taken as Hg(NO ₂) ₂ Grm.	Hg present as Hg(NO ₂) ₂ Grm.	Excess am- monium C.m. ³ .	HNO ₃ at sp. gr. 1.15 C.m. ³ .	Volume at precipitation C.m. ³ .	Hg found. Grm.	Error. Grm.
K.						
0.1217	—	2.4	—	100	0.1217	+0.0000
0.1217	—	2.4	—	100	0.1217	+0.0000
0.1122	—	2.4	—	100	0.1124	+0.0002
L.						
0.1122	0.0067	0.93	2	100	0.1130	+0.0008
0.1122	0.0067	0.93	2	100	0.1112	-0.0010
M.						
0.1122	0.0067	4.40	2	100	0.1124	+0.0002
N.						
0.1122	0.0135	0.72	4	100	0.1125	+0.0003
O.						
0.2244	0.0071	1.68	4	100	0.2253	+0.0009
0.2244	0.0071	2.46	4	100	0.2241	-0.0003
P.						
0.2244	0.0048	0.54	4	200	0.2248	+0.0004
0.2244	0.0048	2.44	4	200	0.2245	+0.0001

In Section K are experiments showing the accuracy of the process where a mercurous salt is precipitated in the absence of a mercuric salt. A small amount of mercuric salt was introduced in experiments L, and an excess of ammonium oxalate in experiment M, a still larger amount of mercuric salt was present in experiment N, and a larger amount of mercurous salt in experiments under O. In experiments in Section P a dilution of 200 c.m.³ was employed both with and without an excess of ammonium oxalate. All the results are within reasonable limits of error.

The work may be summed up briefly as follows:—Mercurous nitrate may be estimated volumetrically by precipitating as the oxalate and determining the excess of the precipitant with permanganate.

The precipitated mercurous oxalate may also be estimated gravimetrically by drying it over sulphuric acid and weighing directly.

In solutions containing 2 to 5 per cent dilute nitric acid, sp. gr. 1.15, mercurous salts may be separated quantitatively as the oxalate from small quantities of mercuric salts.

If about 0.12 grm. of mercury is present as the nitrate in 100 c.m.³ of water, about 12 per cent of that amount of mercury as the mercuric salt may be present without interfering with the accuracy of the estimation, and even 20 per cent may be present before an appreciable rise in the result is apparent. If the amount of mercurous salt present is doubled, the amount of mercuric salt which may be present is cut down about one-half.

The author wishes to thank Professor F. A. Gooch for many kind suggestions given during the course of this work.

NOTICES OF BOOKS.

Domestic Science. The Science of Domestic Economy and Hygiene treated Experimentally. By THOMAS CARTWRIGHT, B.A., B.Sc. (Lond.). With numerous illustrations. London, Edinburgh, and New York: Thomas Nelson and Sons. 1900. Pp. 215.

As its title indicates, this book is more particularly suited for girls; indeed, the author insists that girls should be made to carry out all the experiments herein described with their own hands, as they will by this means more firmly grasp the fundamental ideas of domestic economy.

The subjects dealt with are of a very elementary character, and comprise weighing and measuring, the general effects of heat on matter, cooking, the purification and separation of liquids, the transmission of heat with regard to clothing, experiments with coal, candles, and gas-flames, the properties and characteristics of various waters, hard and soft; the preservation of food, &c.; the whole forming a useful and interesting little volume for school-children of both sexes.

Notes on Five Years' Experiments on Hop-manuring, conducted at Golden Green, Hadlow, Tonbridge. By Dr. BERNARD DYER. Reprinted from *The Agricultural Gazette*. London: Vinton and Co. 1900. Pp. 23.

In this pamphlet Dr. Dyer continues the record of several years' experiments on the manuring of hops, more particularly with the regard to the use of nitrate of soda. On the first page we find the following rather contradictory statements, which we give in the author's own words:—"For many years there has been prevalent among hop-growers an idea that nitrate of soda is an unsafe manure for hops, and likely to injure their quality. On the other hand, the consumption of nitrate of soda in our hop-gardens has, of late, been steadily increasing. Nevertheless, a very large number of hop-growers, while freely using nitrogenous manures of other kinds, fear to make use of nitrate of soda."

From the above statement it would appear that the only explanation of the increased consumption of nitrate of soda would be accounted for by the area under cultivation being increased; this we believe is not the case, but rather the opposite to what has been taking place.

Dr. Dyer's experiments, however, have been carried out with a view to deciding whether or no nitrate of soda has any deleterious effect on the hop crop. It would seem that if nitrate of soda has in some hands proved hurtful, it is because it has been applied without a proper quantity

of phosphates or potash, or because it has been applied too abundantly, or because it has been applied at the wrong time.

The general scheme of the experiments is that on one plot phosphates and potash are applied without any nitrogen, while on five other plots nitrate of soda is added in quantities varying from 2 cwt. up to 10 cwt. per acre, for the most part in successive dressings of 2 cwt. per acre. In the first year the nitrate was not applied till May, while in the succeeding years it was applied earlier, until, in 1899, the first dose was put on early in January.

An examination of the results for this latter year shows that nitrate of soda has proved a valuable addition to the natural resources of the soil, the quantity of hops produced on the nitrated plots being from 2 to 4½ cwt. per acre in excess of the crop produced without artificially applied nitrogen, while the good effect in quality proved to be of even greater importance.

CORRESPONDENCE.

STANDARD OF ATOMIC WEIGHTS.

To the Editor of the Chemical News.

SIR,—Acting upon the suggestion of the International Commission on Atomic Weights I offer the following as good reasons for taking oxygen = 16 as the standard:—

1. The atomic weights more nearly approximate whole numbers as they are now given.

2. For student computations and for most practical purposes scarcely half a dozen of the atomic weights need have any decimal at all, the approximate whole number being sufficiently accurate. With hydrogen as the standard twenty differ as much as 0.4 from whole numbers.

3. With oxygen = 16, hydrogen may still be considered as equal to 1, and the decimal may generally be disregarded.

4. For the teaching of chemistry the approximate whole number is desirable, and the accurate weight need be found only in the table.

Let us have only one standard and avoid the present confusion.—I am, &c.,

J. I. D. HINDS.

University of Nashville,
Nashville, Tennessee, U.S.A.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 13, September 24, 1900.

Nature of the Combustible Gases found in the Air of Paris.—Armand Gautier.—The author's former researches on the combustible gases of the atmosphere were performed on pure air, obtained from places free from animal or vegetable contamination. Such a gas contains 19.5 hundred-thousandths of its volume of free hydrogen; this gas is accompanied by mere traces of hydrocarbons. In the air of towns this hydrogen is mixed with other combustible vapours present in sufficient quantity for estimation. The author examined especially the air of Paris, which no doubt is practically the same as that of other industrial and populous towns, and found that the combustible portion of the atmosphere of the streets has the following mean composition, calculated as parts per 100 litres:—Free hydrogen, 19.5 c.c.; marsh gas, 12.1 c.c.; gases very rich in carbon (benzene and its analogues), 1.7 c.c.; oxide of carbon (with traces of hydrocarbons of the series C_3H_{2n} and C_4H_{2n+2}), 0.2 c.c.

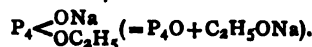
Crystallised Monocalcic Aluminate.—Em. Dufau.—When a mixture of alumina and calcium oxide is heated to a sufficiently high temperature, a combination of the two oxides takes place with formation of monocalcic aluminate, Al_2O_3Ca , crystallised in needle-shaped crystals. This form of crystallisation throws this aluminate out of the group of spinels. It resembles in this manner the aluminate of glucinum, which up to the present time formed the only exception. It may be remarked that calcium chromite, Cr_2O_3Ca , which the author describes in a previous paper, and calcium ferrite, Fe_2O_3Ca , described by J. Percy, also crystallise in needles, and for this reason ought not to be classed as spinels.

MISCELLANEOUS.

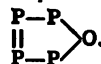
The Lower Oxides of Phosphorus.—A. Michaelis and M. Pitzsch.—A large number of sub-oxides have been described, of which the authors give a detailed history. These compounds contain from 79.49 per cent to 89.08 per cent of phosphorus. They are:—

1. The oxide of Pelouze P_3O
2. The oxide of Le Verrier P_4O
3. The first oxide of Gautier P_3OH
4. The second compound of Gautier .. P_3H_2O
5. The third compound of Gautier .. $P_3H_2O_2$
6. The compound of Franke $P_4H(OH)$
7. The oxide of Besson P_2O

The authors have taken up the analysis of these different compounds. To free them from mixed amorphous phosphorus and hydride of phosphorus, they were, after washing with water and the elimination of ordinary phosphorus, dissolved in alcoholic KOH, their only solvent, and reprecipitated with a dilute acid. The authors have distinctly found that only one sub-oxide exists, that of Le Verrier, P_4O . Le Verrier's process is complicated, and the authors have prepared this body by two different methods. The first consists of treating granulated phosphorus, washed with alcohol, with an aqueous alcoholic solution of KHO; hydrogen and a little phosphoretted hydrogen are given off, and a red solution is obtained, from which acids precipitate the sub-oxide according to the equation $P_4 + H_2O = P_4O + H_2$. The second method consists of dehydrating hypophosphorous acid by means of acetic anhydride. The anhydride P_2O being unstable is decomposed according to the equation $2P_2O = P_4O + O$, which oxygen unites with 1 molecule of PO_2H_3 . The precipitate is washed with water and alcohol, then dried in vacuo over P_2O_5 . The sub-oxide P_4O is a fine powder, orange-red, or yellow in colour according to its state of division, with a density of 1.9123 at 26°; it attracts moisture, and gives off an odour of phosphoretted hydrogen; when moist it ignites at 90°, but when dry it can be heated to a much higher temperature. When heated in an inert atmosphere it splits up according to the equation $5P_4O = P_2O_5 + 18P$. Chlorine transforms it into $POCl_3$ and PCl_5 . If it is moistened it gives PO_2H_3 . Warm SO_2 gives PO_2H_3 and H_2S ; NHO_3 inflames it. By its action many metallic solutions are precipitated in the state of phosphide. The alkalis dissolved in aqueous alcohol dissolve it, giving a deep red coloured solution, possibly the alcoholate,—



This red solution gradually loses its colour, giving off hydrogen and phosphoretted hydrogen, and it then consists of PO_2H_3 ; this is formed according to the following equation:— $P_4O + 7H_2O = 4PO_2H_3 + H_2$. The constitution of P_4O may be represented by—



—*Lieb. Annal. Chem.*, vol. cccx., p. 45.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Estimation of Graphite in Ferro-silicon.—Will some reader kindly inform me of a reliable process for the above.—H. JARVIS, 53, Bannock Street, Sheffield.

MEETINGS FOR THE WEEK.

FRIDAY, 26th.—Physical, 5. Exhibition of Experiments illustrating certain Phenomena of Vision, by Dr. Shelford Bidwell, F.R.S. "Concentration at the Electrode in a Solution, with special reference to the Liberation of Hydrogen by the Electrolysis of a mixture of Copper Sulphate and Sulphuric Acid," by Dr. J. S. Sand. "Electromotive Force and Osmotic Pressure," by Dr. R. A. Lehfeldt.

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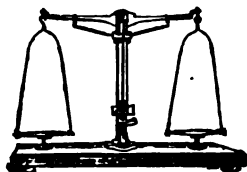
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THE CONSTITUTION OF CAMPHOR.*

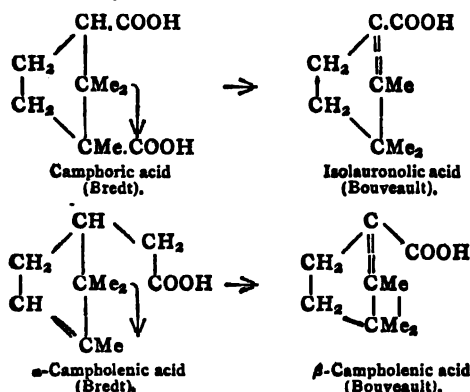
By ARTHUR LAPWORTH, D.Sc.

THE paper deals briefly with the more important facts which have a direct bearing upon the question of the constitution of camphor, and the present position of our knowledge is discussed.

Reasons are given for the view that the Bredt and the Perkin-Bouveault formulæ are the only ones now worthy of consideration, and the evidence in favour of each is reviewed. It is shown that apparently conclusive evidence in favour of either may be brought forward, but that the weight of evidence leans in a marked way towards that of Bredt, the most important obstacles to the final acceptance of that formula being the properties and constitution of the stereoisomeric campholytic and isolaunonic acids and of β -campholenic acid.

The significance of the evidence derived from the study of the β -campholenic derivatives is shown to be materially diminished by the fact that these derivatives are secondary products, being produced, probably in all cases, from the α -derivatives, and the properties of the latter are exactly represented by Bredt's formulæ, the inference being that the passage from the one set to the other is the result of an unusual intramolecular change. Moreover, from a consideration of the inactivity and general properties of β -campholenic acid on the one hand and of isolaunonic acid on the other, it would appear that these two substances are very closely related, the former being probably derived from the latter by the substitution of $-\text{CH}_2\text{COOH}$ for $-\text{COOH}$, so that it may be presumed that the internal changes which result in their formation are very similar.

In the author's opinion, the formulæ for isolaunonic acid and β -campholenic acid suggested by Bouveault are almost certainly correct, and it is equally probable that the compounds from which they are obtained are represented by Bredt's formulæ. A comparison of these structures is made, and it is pointed out that the essential structural change appears in each case to be the migration of a single methyl group from one carbon atom to another immediately adjacent.



Some assumption of this kind would certainly appear to be necessary, and the one here advanced is probably the simplest possible. The migration of methyl groups from one carbon atom to another is not infrequent, and to

recall the readiness with which pinacone is converted into pinacolone is to perceive the possibility of a transference of methyl in the case here considered. It is specially significant, too, that when a methyl group migrates in the molecule of a hexamethylene compound during conversion into a benzenoid derivative, it is the adjacent carbon atom to which it is transferred.

The properties of α -bromocamphoric anhydride and camphanic acid are discussed in connection with the question of the relationship of camphoric acid to succinic and glutaric acid, and reference is also made to the properties of Noyes's hydroxydihydrolauronic acid, which are not in accordance with the requirements of Bredt's formula for camphor.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART IV.

NICKEL AND COBALT.

By HARRY BREARLEY.

(Continued from p. 187).

COBALT.

I. SEPARATING FROM NICKEL.

I. 1. Depending on Behaviour with KCN.—

598. GIBBS (C. N., xi., 125).—By using a solution of HgO in KCN to precipitate Ni from the double cyanide of Ni and Co, the separation is immediate and complete; the precipitates may be ignited and weighed at once; Co determined by difference. Separation with KNO_3 lengthy and difficult.
599. HADOW (C. N., ii., 86).—In Liebig's process KCN should be added before the KHO; SiO_2 should be absent. Precipitation of the Ni by Cl or a salt of Hg preferable to HgO. If cobalt is finally precipitated with HgNO_3 (Wöhler's process), chlorides and sulphates should be absent.
600. FLECK (C. N., xiii., 299).— NH_3 solution exposed to air, $(\text{NH}_4)_2\text{S}$ added, NH_3 evaporated, and KCN added; the nickel sulphide dissolves.
601. GUYARD (C. N., xxxiv., 255).—Precipitate as sulphides, dissolve out Ni with KCN, acidulate filtrate, and ignite precipitated nickel cyanide to oxide.
602. JORISSEN (C. N., xlv., 240).—Precipitate with NaHO and Br, and dissolve out Ni with KCN; not good for much Ni and little Co.

I. 2. Depending on Behaviour of Oxidised Compounds.—

603. FLEISCHER (C. N., xxii., 96).—Add hypochlorite and KHO, boil until precipitate is black, then dissolve out Ni_2O_3 with $(\text{NH}_4)\text{HO}$.
604. DONATH (C. N., xlvii., 248).—Halve the solution; precipitate both metals as sesquioxides in one, and Co alone with KHO and I in the other; distil each precipitate with HCl and estimate the Cl with KI and hyposulphite.
605. DELVAUX (C. N., xliii., 196).—Dissolve oxides or sulphides in aqua regia, add $(\text{NH}_4)\text{HO}$ and permanganate, then KHO to precipitate the Ni. The precipitate must be separated from Mn.
606. VORTMANN (C. N., xlix., 150).—Uses hypochlorite instead of permanganate (605); detects traces of either metal in the other. (See also 510).
607. McCULLOCH (C. N., lvi., 27).—Examination of Fleischer (603) and Donath (604) processes. The Co is not oxidised to Co_2O_3 . Nickel salts precipitated with NaHO and an oxidising reagent varied from Ni_2O_3 to Ni_3O_4 .
608. BAYLEY (C. N., xxxix., 81).—Ni and Co are not oxidised by hypochlorite to Ni_2O_3 , but to Ni_3O_4 and Co_3O_5 , which are further decomposed on boiling.

* Abstract of a Paper read before the British Association (Section B), Bradford Meeting, 1900.

609. ARNOLD ("Steel Works Analysis," p. 166).—By precipitating with Br and NaHO, the compound formed contained only about 15 per cent Ni_2O_3 .
610. CARNOT (C. N., liz., 183).—Co precipitated by H_2O_2 and KHO is exactly Co_2O_3 ; precipitated by hypochlorite, Br, or I, the proportion of O is greater; under the latter conditions, the Ni is exactly Ni_2O_3 . Estimation and separation of the metals based on these facts. McCulloch (C. N., liz., 205) disputes the fundamental facts, and (C. N., liz., 208) makes unsuccessful attempts to separate the metals after oxidation in ammoniacal solution.
611. FISCHER (Z. C. S., lvi., 653).—Alkaline solution boiled with H_2O_2 , and the oxygen in the Co_2O_3 estimated with KI and $\text{Na}_2\text{S}_2\text{O}_3$.
612. CARNOT (C. N., lx., 54).—To acid solution add $(\text{NH}_4)\text{HO}$ and H_2O_2 ; or precipitate ammonio-cobalt molybdate from acetic solution with $(\text{NH}_4)_2\text{MoO}_4$; Ni in solution; Zn and Cu do not interfere. A good way of detecting cobalt.
613. TERRELL (C. N., xliii., 133).— KMnO_4 added to hot ammoniacal solution, and then a faint excess of HCl; after a time, roseo-cobaltic hydrochlorate is precipitated. Detects 0.01 per cent Co in Ni.
- I. 3. Separation as Phosphate.—
614. DIRVELL (C. N., xl., 268).—Co precipitated by microcosmic salt and ammonium acetate as $\text{NH}_4\text{O} \cdot 2\text{CoO} \cdot \text{PO}_3 + 2\text{H}_2\text{O}$ and ignited to pyrophosphate. The method is rapid.
615. HOPE (Z. S. C. I., 1890, 375).—Uses this process with electro-estimation for analysis of ores, mattes, &c.
616. CLARKE (C. N., xlviii., 262).—A careful examination of Dirvell's process. Accurate results with any proportion of Co and Ni. Process considerably modified.
617. CLARKE (Z. S. C. I., 1896, 867).—Dirvell's process does not precipitate Co if present as cobaltic salt, and therefore by means of Br and H_2O_2 , and the process (575) Ni is precipitated in the presence of Co.
- I. 4. Miscellaneous Processes for Separating Ni and Co.—
618. FLEITMANN (C. N., xxxii., 260).— KNO_3 fails to separate 1 per cent Co from Ni. The Co is precipitated along with a little Ni by means of hypochlorite, and then the nitrite separation of the Co made.
619. BAUBIGNY (C. N., lxiv., 224).—Ba, Ca, Sr, and Pb interfere with the nitrite separation. KNO_3 should always be tested for Pb.
620. ILINSKI and KNORRE (C. N., li., 170).—Precipitate cobalt with nitroso- β -naphthol, ignite with oxalic acid, and reduce to metal; Fe and Cr must be absent (see 25 and 26).
- C. N., lii., 301.—Cobalti-nitroso- β -naphthol is permanent in the presence of acids, alkalis, and oxidising and reducing agents; stability with various reagents, and means of preparing the nitroso- β -naphthol given.
621. KNORRE (Z. C. S., xlviii., 840; and lxiv., li., 500).—Scheme for analysis of commercial Ni; separation as above; cobalt ignited to Co_2O_3 .
622. PHIPSON (C. N., xxxvi., 150).—Co and Ni are precipitated together by potassium xanthate; on making slightly ammoniacal, the Ni passes into solution.
623. HAVENS (C. N., lxxviii., 323).—Pinner's process (512) does not effect a complete separation. Part of the cobalt, if present in considerable amount, goes down with the Ni.
624. GUCCI (Z. C. S., l., 1077).—Mixed sulphides are fused with KNO_3 ; from the oxides, undissolved by water leaching, Ni is completely dissolved by 1.20 HNO_3 .
625. BAUBIGNY (C. N., lviii., 55).— H_2S does not precipitate all the Co before acting on the Ni, in an acetic solution, as has been stated, and therefore no process on these lines is quantitatively available. (See 561).
626. CLARKE (C. N., xx., 154).—Metals precipitated with ferricyanide from a $(\text{NH}_4)\text{Cl}$ solution; nickel dissolved from precipitate with $(\text{NH}_4)\text{HO}$; cobalt absolutely insoluble; process given as qualitative.
627. HERRENSCHMIDT (C. N., lxix., 112, &c.).—Cyanide superior to nitrite method of separation. Traces of Ni can be microscopically detected in cobalt hydroxide; weigh Ni and Co together as sulphates; determine Ni separately as sulphate after separating with KCN, Br, and Cl; Co by difference. Discusses (p. 128) Baubigny's separation of Zn (534); review experiments on the state of oxidation when Ni and Co are treated with oxidants in alkaline solution. Carbon monoxide (Mond's process) is recommended for the absolute separation of Ni and Co.
628. KRAUSS (C. N., lxiii., 254, &c.).—An examination of fourteen methods founded on the different behaviour of the higher oxides of the metals; two depending on the behaviour of the sulphides, Dirvell's process (614) and Clarke's modification of it (616); and six methods based on various principles. This memoir practically covers the whole ground up to 1891.
- II. SEPARATION OF COBALT FROM OTHER ELEMENTS.
629. Zinc.—VORTMANN (Z. C. S., lxviii., ii., 89).—Electrodeposition of Co from alkaline tartrate solution.
630. WALLER (Z. S. C. I., 1897, 1043).—A summary of proposed means of making the electro-separation. (See also 524, 528, &c.).
631. Bismuth.—JANNASCH and KAMMERER (C. N., lxix., 91).—On pouring a nitric acid solution of the metals into ammoniacal H_2O_2 Bi is precipitated.
- III. GRAVIMETRIC ESTIMATION OF COBALT.
632. GIBBS (C. N., xvii., 172).—Neutral solution boiled with soda acetate and hypochlorite. The Co_2O_3 , reduced to Co in H, is free from alkali; Ni may be similarly estimated; this is Popp's process.
- 632a. GIBBS (C. N., xxviii., 51).—Co converted to cobalticyanide, precipitated with HgNO_3 and HgO , ignited in air, and then reduced to metal in H.
633. RUSSELL (C. N., vii., 43).—Ignition in a powerful blast blowpipe and cooling in a current of CO_2 forms true protoxide of cobalt.
634. SALVETAT (C. N., x., 91).—Oxide is calcined with a salt of Al, leaving a known residue. The increase in weight represents protoxide of cobalt.
635. FRESSENIUS (C. N., iv., 150).— $(\text{NH}_4)_2\text{S}$ does not precipitate dilute Co solutions; NH_4Cl assists, $(\text{NH}_4)\text{HO}$ slightly retards, precipitation.
636. MOORE (C. N., lxx., 75).—Decompose Mn ores by fusion; precipitate Ni and Co as sulphides, convert to sulphates, and electro-deposit; separate Co by precipitation as ammoniacal phosphate. A rapid process is based on the absorption of oxygen by ammoniacal solutions of cobalt according to the equation $2\text{CoO} + \text{O} = \text{Co}_2\text{O}_3$.
- IV. VOLUMETRIC ESTIMATION OF COBALT.
- IV. 1. Oxidimetric Processes.—
637. WINKLER (C. N., x., 215).—Co may be estimated in presence of Ni by precipitating with HgO and a standard solution of KMnO_4 ; recognises 0.1 per cent Co in Ni; oxygen compounds of Cl, S, As, and P interfere; As and P eliminated by adding Fe_2Cl_6 and precipitating with HgO .
638. DONATH (C. N., xli., 15).—Modified Fleischer's process (603). In one portion converts to Co_2O_3 and Ni_2O_3 with bromine, and in another to Co_2O_3 and NiO with KHO and boiling. Each precipitate is distilled with HCl.
639. MCCULLOCH (C. N., liz., 51).—Measures the oxygen necessary to convert cobalto- to cobalti-cyanide;

- the operation is performed in absence of air and at barometric pressure. Ni, Mn, Pb, P, As, Al, Zn, Sb, Mo, and Ur do not interfere; Cu and Fe do.
640. MOORE (C. N., lxxviii., 295).—The change from cobalto- to cobalti-cyanide either in alkaline or acid solutions is accompanied by greater absorption of O than is expressed by the usual equation, $2K_4CoCy_6 + H_2O + O = K_6Co_2Cy_{12} + 2KHO + H_2O$.
641. JOB (C. N., lxxviii., 254).—Alkaline cobalt solutions peroxidised with H_2O_2 , and the Co_2O_3 formed titrated with a ferrous solution.
642. KARSLAKE (J. C. S., lxi., ii., 194).—Precipitate as cobaltinitrite, boil with KHO, and titrate with permanganate; the oxidation is from the nitrous acid of the $K_2Co(NO_2)_6$ to nitric acid.
643. JOLLES (J. C. S., lvi., 798).—The cobaltous solution is run into a measured volume of manganate until the latter is decolorised; $CoMnO_3$ is precipitated.
644. REIS and WIGBERT (J. C. S., lx., 620).—Oxidation to cobaltic oxide with permanganate in presence of ZnO ; excess of $KMnO_4$ titrated with arsenious acid.
645. PURGOTTI (J. C. S., lxxii., ii., 77).—After converting to the sesquioxides, either Ni or Co may be estimated by titrating with a solution of the blue oxide of molybdenum (Mo_3O_8). (See also C. N., xxxviii., 22).
646. ROSSLER (C. N., xli., 184).—Cobaltous solution mixed with standard silver and KHO solution; $Ag_4OCO_2O_3$ is precipitated. Proceed thence as for Mn (207). Ni causes low results.

IV. 2. Miscellaneous Processes.—

647. NEUMANN (J. C. S., lxxviii., ii., 64).—Either Ni or Co estimated by titration with soda sulphide.
648. KRIEGER (J. C. S., lxxviii., ii., 534).—The HCl solution of the ore precipitated with chalk, and a fraction of the filtrate colorimetrically compared with standard cobaltous chloride solution.
649. BREARLEY (C. N., lxxvi., 165).—Cyanometric estimation, using the AgI indicator as for Ni (581).
650. HARRIS (J. C. S., lxxiv., ii., 487).—A criticism of Winkler's (637), McCulloch's (639), Fleischer's (603), Donath's (638), and von Reis's methods. None of them quite satisfactory.

V. DETECTION OF COBALT.

651. BRAUN (C. N., xli., 58).— KNO_3 and acetic acid added to a solution of Co in KCN gives a deep red solution.
652. SKEY (C. N., xv., 111).—In an ammoniacal solution in tartaric or citric acid ferricyanide produces a dark red colour. Can detect one part in 400,000. C. N., xv., 328.—Any other acid may be substituted for tartaric (see 588).
653. REICHEL (C. N., xliii., 7).—The KHO precipitate of the Ni and Co is heated with KHO (solid) and a little H_2O . The Co dissolves with a blue colour, and may be precipitated from the filtrate with ether.
654. TATTERSALL (C. N., xxxix., 66).—Add KCN and yellow $(NH_4)_2S$; blood red = Co; only Cu interferes. Papasogli (C. N., xxxix., 149) says this test is most sensitive when the $(NH_4)_2S$ is superstratified.
655. PAPASOGLI (C. N., xli., 74).—The previous reaction and a red colour formed when Zn is added to the double Ni and K cyanide are used to detect the one metal in the presence of the other.
656. PAPASOGLI (J. S. C. I., 1899, 74).—Saccharose and soda give a violet colour with one part Co in 50,000; further tests with amylsulphocarbonate, &c., are applied when Ni is also present.
657. MOORE (C. N., lxxviii., 295; and lxxi., 81).—Investigates the cause of the red colour used by Papasogli (655) as a test for Ni.
658. BALL (C. N., lxx., 36).—Pour a little of the solution

on to anhydrous thiosulphate and heat; green colour = Co. Ni, Fe, Mn, &c., do not interfere.

659. DURRANT (C. N., lxxiii., 228).— $Na_2CO_3 + H_2O_2$ give a green liquid with Co in presence of much Ni.
660. COHEN (J. S. C. I., 1898, 605).—Co is detected in presence of Ni by its deposition in the fine cracks of a glass tube (stenolysis).
661. VON ILINSKI (J. C. S., lxx., ii., 451).—Add fresh nitroso- β -naphthol to HCl solution; dark red precipitate = Co. Works well in the presence of Ni.
662. WOLFF (C. N., xlvii., 94).—Fe precipitated from solution of the sulphocyanides with Na_2CO_3 . Shake the filtrate with amyl alcohol and ether; cobalt is distinguished with the spectroscope by the absorption band between C and D.
663. BETTING (J. S. C. I., 1899, 519).—To detect Co in Fe treat the solution with KCNS and $Na_2S_2O_3$ until the red colour disappears, filter, and add alcohol-ether; a blue colour indicates Co.
664. JAWOROWSKI (J. C. S., lxxv., 61).—Add soda pyrophosphate to neutral solution, dilute till solution is colourless, add Na_2CO_3 and Br; a reflected green colour indicates cobalt.

PREPARATION AND PROPERTIES OF THE SO-CALLED "NITROGEN IODIDE."

By F. D. CHATTAWAY and E. J. P. ORTON.

NITROGEN iodide was originally obtained by Courtois (*Ann. Chim.*, lxxxviii., 304) in 1813 by the direct action of ammonia on solid iodine. Solutions of iodine in alcohol, chloroform, carbon tetrachloride, or aqueous potassium iodide, were substituted for solid iodine by later observers, and both alcoholic and aqueous solutions of ammonia have been employed. In all cases a black or dark brown amorphous solution was obtained.

Sérullas (*Ann. Chim. Phys.*, 1825 [2], xxii., 172; and 1829, xlii., 200) prepared the substance by the interaction of iodine monochloride and a solution of ammonia, a method which was afterwards employed by Bunsen (*Liebig's Ann. Chem.*, 1852, lxxxiv., 1). We have prepared nitrogen iodide by these various methods, and have compared the products, which we find to be identical when proper precautions are taken to ensure the removal of all free iodine and ammonia and to prevent decomposition.

Whenever iodine itself is used, whether in solution or as a solid, less than half appears as nitrogen iodide. Exact experiment showed that at ordinary laboratory temperatures the nitrogen iodide formed contains about 47.5 per cent of the iodine used. Of the remainder, 51.6 per cent appears as ammonium iodide and 0.8 per cent as ammonium iodate.

The use of alcoholic solutions of iodine or ammonia decreases largely the yield, because the nitrogen iodide reacts rapidly with the alcohol to form iodoform, which, moreover, can never be wholly removed from the product. Bunsen, however, used this method and obtained a substance, analysis of which led him to the formula $N_2H_5I_3$ for nitrogen iodide. All our analyses of nitrogen iodide prepared in various ways agree absolutely with this formula.*

Preparation of Nitrogen Iodide by the Action of Iodine Monochloride on a Solution of Ammonia.

When iodine monochloride is used 95 per cent of the iodine appears as nitrogen iodide. The remainder is converted into ammonium iodide and iodate, while the chlorine appears as ammonium chloride. We have carefully investigated this method, and consider it the most

* Bunsen determines the relation of nitrogen to iodine only, and in his method of analysis any trace of iodoform present would not cause error.

suitable for preparing pure nitrogen iodide in large quantities. The best procedure for the preparation of the iodine monochloride and of the nitrogen iodide is as follows:—

One hundred grms. of finely powdered iodine are placed in 300 c.c. of hydrochloric acid of sp. gr. 1.15 in a porcelain basin, and 28 c.c. of nitric acid of sp. gr. 1.41 are added. This quantity of nitric acid provides just sufficient chlorine to convert the iodine into iodine monochloride. The mixture is warmed on a water-bath to about 40°, and continuously stirred, the beginning of the reaction being marked by the colour of the liquid changing from brown to pale yellow. The iodine gradually dissolves, and the solution becomes orange in colour. If the mixture be well stirred, and the temperature not allowed to rise above 40°, no chlorine escapes. After the whole of the iodine has dissolved, the water in the bath is boiled for some time in order to expel the nitrosyl chloride. With the excess of hydrochloric acid used, the solution of iodine monochloride is perfectly stable and undergoes no decomposition even when boiled. To prepare nitrogen iodide it is cooled and diluted by adding about three times its bulk of crushed ice.

For every 10 grms. of iodine 100 c.c. of strong commercial ammonia (sp. gr. 0.880) are poured over about three times their weight of crushed ice, and the cold solution of iodine monochloride slowly run in, the mixture being vigorously stirred during the addition.* The black precipitate of nitrogen iodide which at once separates is filtered off by a pump through asbestos and washed with dilute ammonia, and finally, if required free from ammonia, three or four times with water. In this way a kilogram of nitrogen iodide can be obtained as a compact cake with perfect safety. It is best, if possible, to leave the solid mass damp with strong ammonia, for then filter-paper can be used instead of asbestos, and the slight decompositions which take place in the total absence of ammonia, and may give rise to local explosions, is prevented.†

Preparation of Crystalline Nitrogen Iodide.

In the course of this investigation it became obvious that nitrogen iodide is not formed by a direct substitution of iodine for hydrogen in ammonia, but that the iodine reacts with ammonium hydroxide, as with other alkalis, to form ammonium iodide and hypiodite, and that the latter then decomposes, forming nitrogen iodide. This view was originally offered by Schönbein (*Journ. Prakt. Chem.*, 1861, lxxiv., 385) as a suggestion, which has been endorsed by Seliwanow (*Ber. d. Chem. Ges.*, 1894, xxvii., 1012). The addition of ammonia to an alkaline solution of potassium hypiodite should, therefore, lead to the formation of NH_4OI , ammonium hypiodite, which should then decompose, producing nitrogen iodide. This we have found to be the case; and, further, under certain conditions of concentration the nitrogen iodide separates in a crystalline form.

The following method gives good results:—A decinormal solution of iodine monochloride is prepared by diluting with water to 1 litre a solution of iodine monochloride, ICl , made as above, from 12.7 grms. of iodine. This dilute solution is unstable, and should only be prepared immediately before use. Fifteen c.c. of this solution are added to 100 c.c. of a half-normal solution of potassium hydroxide (3 per cent), and then, as rapidly as possible, 10 c.c. of ammonia (sp. gr. 0.880) are run in while the solution is gently shaken. The pale yellow liquid remains clear for a short time, but within a minute glittering copper-coloured crystals of nitrogen iodide begin to sepa-

rate, and after a few minutes the crystallisation is complete. The yield is satisfactory, being from 65 to 70 grms. per 100 grms. of iodine. When larger quantities than those indicated are used the yield is not so good, owing to the difficulty of mixing the solutions sufficiently rapidly. Under the microscope very minute needles first become visible, which steadily grow to the usual crystals. The large excess of potassium hydroxide used prevents any setting free of iodine when the acid solution of iodine monochloride is added to it. If more than 15 c.c. of ICl is added to 100 c.c. KOH , the crystals separate more rapidly and are smaller. With further increased concentration the crystals become mixed with amorphous nitrogen iodide, which finally forms the chief product.

Replacement of ammonia by a dilute solution of an ammonium salt also causes a deposition of crystalline nitrogen iodide, if the ammonium salt be added very cautiously. An amorphous precipitate is obtained if the solution of ammonium salt be added rapidly, or if it be concentrated.

Amorphous nitrogen iodide can be converted into the crystalline variety by an apparent re-crystallisation from a hot solution of ammonia. The conversion is, however, really due to the occurrence of a reversible reaction in the system (nitrogen iodide + ammonium hypiodite + ammonium hydroxide). In this system, at the state of equilibrium, the concentration of ammonium hypiodite is greater at a high than at a low temperature with a given concentration of ammonia. To obtain crystalline nitrogen iodide by this method about 0.5 gm. of the amorphous substance is heated with 100 c.c. of thrice-normal ammonia solution, in which it partially dissolves, producing a pale yellow solution, and this, after filtration through asbestos, deposits crystals when rapidly cooled. With larger quantities the time required to cool the bulk of hot liquid leads to the conversion of so large a proportion of the ammonium hypiodite into iodate and iodide that little nitrogen iodide separates.

Crystals can also be obtained by the direct addition of a solution of iodine monochloride to ammonia. For this purpose the solution of ICl must be about one-fiftieth normal, and must be added very slowly to a fairly concentrated solution of ammonia.*

Properties of Nitrogen Iodide.

Crystals of nitrogen iodide suspended in water look like splinters of burnished copper, and when dry have a ruby-red colour and a fine lustre. Ordinary amorphous nitrogen iodide shows no trace of crystalline structure, and appears quite black when suspended in water, but if filtered off and dried is seen somewhat to resemble the crystalline substance in colour.

Pure nitrogen iodide is without effect on a neutral solution of litmus, and gives no reaction of iodine when shaken with chloroform. In contact with water it soon shows signs of decomposition, the amorphous more rapidly than the crystalline variety. The crystals lose their lustre, and under the microscope are seen to be etched and corroded. Too prolonged washing with water on the filter will cause this decomposition to take place. Free iodine is then always found by the chloroform test in the solid residue, while free ammonia can be detected in the filtrate.†

Nitrogen iodide can be dried over lime or baryta in an

* The solution of ammonia should not be added to the solution of iodine monochloride, for then iodine is liberated and the yield much reduced.

† André (*Journ. Pharm.*, 1896, xlii., 137) obtained nitrogen iodide by the addition of ammonia to a solution of iodic acid in hydrochloric acid. Such a solution contains iodine monochloride after it has been heated, for, as is well known, iodic and hydrochloric acids then react according to the equation $\text{HIO}_3 + 5\text{HCl} = \text{ICl} + 2\text{Cl}_2 + 3\text{H}_2\text{O}$.

* The formation of nitrogen iodide has been observed in the action of bleaching-powder on a solution of ammonium iodide.—(Playfair in discussion on Gladstone's paper, *Chem. Soc. Journ.*, 1852, iv., 34). In this case undoubtedly ammonium hypiodite is first produced, and from it the nitrogen iodide is formed. The product separating is always mixed with calcium iodate, which can only with difficulty be removed by prolonged washing with ammonia. In the electrolysis of an ammoniacal solution of potassium iodide, Loosnitch and Jowitschitch (*Ber. d. Chem. Ges.*, xlix., 2430) noticed that at the positive pole nitrogen iodide was deposited, and that hypiodite could be recognised in the solution.

† Pure nitrogen iodide consequently cannot be obtained, as many observers have stated, by washing the product until the filtrate becomes neutral.

atmosphere of ammonia, if light be absolutely excluded, without any decomposition taking place. When dry it can be safely detached from a porous tile with a spatula, but slight percussion, pressure between hard surfaces, or rapid heating cause it to detonate with violence. The whole mass explodes at once without scattering, but the explosion is never communicated to any of the substance lying only a few centimetres away. A puff of violet vapour surrounded by a cloud of white fumes is seen, and in a dark room a green flash of light is noticed, resembling in colour the flame of burning ammonia. Nitrogen iodide is remarkably sensitive to light. Bubbles of nitrogen are slowly given off in diffused light from the compound suspended in water, while in direct sunlight rapid effervescence takes place. The dry substance in diffused light becomes gradually covered with minute crystals of iodine, which appear more quickly and grow more rapidly in sunlight.

Partially decomposed nitrogen iodide is very unstable and explodes at the slightest touch.

The following description of the crystals of nitrogen iodide has been given us by Mr. W. J. Pope, who very kindly undertook their examination:—The crystals are small, flattened needles, of a bright ruby colour in transmitted light. The extinction through all faces in the zone of the long edge is straight, and the crystals are probably orthorhombic; the forms would then be $\{001\}$, $\{101\}$ and $\{110\}$ and the crystals are lengthened in the direction of the axis b . The plane angle between the edges $001 : 101$ and $001 : 110$ is 140° , and very frequently only one face of $\{110\}$ is present at one end. An optic axis can be sometimes just discerned at the edge of the field, emerging in the plane $\{010\}$.

The crystals are dichroic. On looking through $\{001\}$ using light polarised in the plane $\{100\}$, light of a beetle-green colour is transmitted, but if the plane of polarisation be $\{010\}$ light of a ruby-red colour comes through.

The specific gravity of crystalline nitrogen iodide is about 3.5. This number has been obtained by drying a quantity of the substance in an atmosphere of ammonia in a specific gravity bottle, and weighing it first in air and then under water.—*American Chemical Journal*, xxiii., No. 5.

THE PREPARATION OF PURE TELLURIUM.*

By JAMES F. NORRIS, HENRY FAY, and D. W. EDGERLY.

In testing a volumetric method (*Amer. Chem. Journ.*, xx., 278), for the estimation of tellurium which we proposed some time ago, we were led to study the methods which had been used for the purification of tellurium. As it appeared that a substance of undoubted purity could be obtained only by a very long series of operations, which involved fusion with potassium cyanide, precipitation, and subsequent distillation in hydrogen, a new method was sought. It seemed probable that the basic nitrate of tellurium might be used for this purpose, as it is a well-crystallised compound, very easily obtained. We accordingly prepared it, and having found that the sample did not agree in some of its properties with those which had been assigned to it in the literature, a careful study of it was made. Klein and Morel (*Bull. Soc. Chim.*, [2], xliii., 198), prepared the compound by dissolving tellurium in nitric acid (1.25 sp. gr.) and evaporating the solution. To the crystals which formed they assigned the formula $4\text{TeO}_2 \cdot \text{N}_2\text{O}_5 \cdot 1\frac{1}{2}\text{H}_2\text{O}$. Only very small crystals were

formed and these were hygroscopic. The nitrate prepared by us by the method of Klein and Morel was obtained in crystals sometimes a centimetre in length and were not hygroscopic. An analysis was accordingly made, which showed that the compound has the formula $4\text{TeO}_2 \cdot \text{N}_2\text{O}_5 \cdot \text{H}_2\text{O}$, or better $\text{Te}_2\text{O}_3(\text{OH})\text{NO}_3$, since the water is not present as water of crystallisation.

In the preparation of the nitrate it is crystallised from nitric acid, and as the impurities present in the tellurium do not form crystalline compounds under these conditions, it was thought that the salt could be obtained quite pure. The tellurium obtained from it would, as a result, be free from those substances with which it is invariably mixed when precipitated from solution by sulphur dioxide or other reducing agents. A sample of basic nitrate was recrystallised three times and then subjected to a careful study, with the result that no impurity was discovered.

On account of the fact that the most trustworthy atomic weight determinations of tellurium have given results which place it in the eighth group in the periodic system of the elements, notwithstanding its striking similarity to sulphur and selenium, the hypothesis has been put forward that tellurium is a mixture of true tellurium with an atomic weight of about 125, and another element with a higher atomic weight. Brauner (*Journ. Chem. Soc.*, lxx., 382; and lxxvii., 549), who spent a number of years studying tellurium, has expressed this as his opinion.

As no known substance was found in the tellurium obtained from the pure nitrate, the element was subjected to a fractionation to determine whether it could be broken down into two or more substances by this means, which proved so effective in the case of didymium. The double bromide of tellurium and potassium made from tellurium dioxide, hydrobromic acid, and potassium bromide prepared with the greatest care, was carried through a fractionation which involved over 200 crystallisations. In order to determine whether this fractionation had accomplished any decomposition, the tellurium from the end fractions was converted into the nitrate, and the loss in weight of the latter compound on ignition determined. Any change in atomic weight could be determined in this way. The results obtained with the two fractions were identical within the limits of accuracy of the method. The differences obtained, 0.4 of a unit in the atomic weight, was probably due to inaccuracies in the method. The conversion of the nitrate into the oxide, as at present carried out, does not appear to give results from which the atomic weight of tellurium can be determined with great accuracy.

Preparation of Basic Tellurium Nitrate.

The tellurium used was obtained from a residue obtained in the electrolytic refining of copper. This residue was a solution which consisted principally of the sodium salts of tellurous, selenous, and silicic acids, with the last-named acid in great excess. Whitehead (*Journ. Amer. Chem. Soc.*, xvii., 849), has described the process by which the copper is refined, and how the liquid is obtained. Tellurium was obtained from this liquid in two ways. At first the solution was diluted with water, and neutralised with sulphuric acid, which threw out a heavy white precipitate of tellurous acid and silica. This mixture was evaporated to dryness twice with hydrochloric acid to render the silica insoluble, and the residue extracted several times with strong hydrochloric acid. From the solution of the chloride so formed, the tellurium was obtained by precipitation with acid sodium sulphite. In later experiments the tellurium was precipitated from the hot alkaline solution with commercial glucose. The tellurium obtained by both methods contained silica and other impurities. The crude metal was added to warm dilute nitric acid (sp. gr. 1.25) and the resulting solution evaporated to dryness in order to insure complete removal of silica. The mixture of basic nitrate and oxide was ignited till free from nitric acid, and was then extracted a number of times with strong hydrochloric acid. The solu-

* Contributions from the Chemical Laboratories of the Massachusetts Institute of Technology. From the *American Chemical Journal*, vol. xxiii., No. 2.

tion in hydrochloric acid was filtered through asbestos, and precipitated with acid sodium sulphite. The sulphite was added slowly until the precipitate formed was black, thus showing that most of the selenium was precipitated. After the mixed precipitates of selenium and tellurium had settled, the solution was decanted, filtered, and the precipitation continued. The tellurium which had been freed from a large share of its impurities by this second precipitation was again dissolved in nitric acid (sp. gr. 1.25), and the basic nitrate obtained from the solution by crystallisation. The nitrate was twice re-crystallised. This was best done as follows:—The salt was stirred with a large amount of nitric acid (sp. gr. 1.25), heated to about 70° C. At a higher temperature the nitrate was decomposed into a mixture of amorphous and crystalline oxide, which was not readily dissolved by the acid. The solution was filtered through asbestos, and evaporated at a temperature of about 80°. In some cases the solution was allowed to cool after crystals appeared. In other cases the evaporation was continued while the crystals were separating from the liquid. By the latter method larger crystals were obtained, which were at times 0.5 c.m. in length. The nitrate crystallises in orthorhombic prisms, terminated by macrodomes and truncated by well-developed macropinacoids and small brachypinacoids. The salt is not hygroscopic, some crystals having stood in the open air for over a month without losing their bright lustre.

Purity of the Basic Nitrate of Tellurium.

The purity of the basic nitrate of tellurium, which had been twice re-crystallised, was studied. It was again re-crystallised from nitric acid, and a qualitative analysis of the crystals was made. The mother-liquor was evaporated to dryness, and also analysed. The presence of no foreign element was detected in either case. Whitehead (*Journ. Amer. Chem. Soc.*, xvii., 849), and Keller (*Journ. Amer. Chem. Soc.*, xix., 778), have determined what substances are present in the crude copper from which the tellurium used in this work was obtained. These are silver, gold, bismuth, arsenic, antimony, and selenium. As none of these elements forms a crystalline compound which is difficultly soluble in nitric acid, it is seen that the basic nitrate of tellurium was readily obtained in pure condition. Brauner has shown that pure tellurium cannot be obtained by precipitation, and that repeated distillation of the metal is necessary to free it from the heavy metals.

In order to test for minute traces of selenium, a common impurity in tellurium, the following method was devised, based on the difference in behaviour of the oxides of selenium and tellurium with hydriodic acid. The former oxide is reduced and iodine set free, while the latter is converted into the tetraiodide. The test is made in this way:—About 0.15 to 0.2 grm. of the oxide to be tested is dissolved in 2 c.c. of a 10 per cent solution of sodium hydroxide, and 3 c.c. of hydrochloric acid (sp. gr. 1.12) is added. The solution is then cooled to the room temperature, carbon bisulphide and 2 drops of a dilute solution of potassium iodide (2 grms. in 100 c.c. water) are added, and the tube is shaken. Under the above conditions, if selenium is present, the carbon bisulphide will be coloured by the iodine liberated, and the small amount of tellurium tetraiodide formed will remain in solution. It is necessary to avoid a large amount of potassium iodide in order to prevent the formation of much tellurium tetraiodide, which would dissolve in the carbon bisulphide, and so obscure the colour of the iodine. Any doubt whether the colour is due to liberated iodine or to tellurium tetraiodide, can be decided definitely by shaking the carbon bisulphide with water. The colour produced by the iodine is not altered while the tellurium tetraiodide is decomposed, and the carbon bisulphide again becomes colourless. The accuracy of the method was tested by mixing known amounts of selenium dioxide with the tellurium to be tested. Using the amounts given above, 0.0012 m.grm. selenium can be detected in the presence of 0.160 grm. of

tellurium dioxide, that is about 1 part in 150,000. No selenium could be detected in the oxide obtained from the pure nitrate when this very delicate test was applied.

It is of interest to note here that a strong solution of potassium iodide acidified with hydrochloric acid, is a very delicate test for tellurium. The dark colour produced is quite characteristic, resembling somewhat in strong solution the colour of platinum solutions containing iodides. The difference is readily shown, however, by dilution, when the colour produced by platinum persists as a pink, while the colour produced by tellurium disappears on account of the decomposition of the iodide.

No foreign substance was found in the tellurium from the nitrate by careful qualitative analysis, yet it was subjected to additional tests. Brauner and Wills found that the purest tellurium obtained by precipitation invariably left a residue when distilled in hydrogen. A sample of the oxide prepared from the nitrate was dissolved in pure hydrochloric acid, and the tellurium precipitated with sulphur dioxide, and washed until free from hydrochloric acid. The tellurium was then distilled in a stream of hydrogen, which was prepared by the action of sulphuric acid on zinc. The gas was purified by the method used by Wills (*Journ. Chem. Soc.*, xxv., 704), in preparing pure tellurium for a determination of its atomic weight. After the distillation a bright stain was left on the porcelain boat. The tellurium was re-distilled a number of times with the same result. The gray, shiny spot on the boat was not affected by hot sodium hydroxide, hydrochloric or nitric acid, and was not volatilised by the heat of a blast-lamp.

From the following experiments it was shown that this residue was due to the union of a small amount of tellurium with the porcelain of the boat at the high temperature required for the distillation. Some tellurium prepared from the nitrate was next distilled in a vacuum. After the first distillation a light gray substance was left in the boat. This was flocculent, dissolved completely in dilute hydrochloric acid and sodium hydroxide, and evidently was tellurium dioxide, which is always present in precipitated tellurium. The tellurium, when re-distilled in vacuum, left no residue, but when distilled in hydrogen a stain was left on the boat as before. Some pure tellurium, obtained by distillation in a vacuum, was heated on porcelain in a blast-lamp flame. A stain was left which was caused by the combination of a small amount of the tellurium with the porcelain. This does not take place when the metal is distilled in a vacuum, as the temperature of distillation is lower.

The oxide prepared from the nitrate was also completely volatile without leaving a residue. It was heated to redness on a platinum foil placed inside of a porcelain crucible. The above experiments show that a careful examination of the tellurium obtained by re-crystallisation of the basic nitrate did not show the presence of any known substance.

Analysis of Basic Tellurium Nitrate.

As the basic nitrate obtained by crystallisation from nitric acid is a stable substance, and is not hygroscopic as Klein and Morel have reported, a careful analysis of it was considered necessary. The water was determined by igniting the substance, which had been previously dried at 120°, in a current of oxygen for two hours. In the front part of the tube was placed metallic copper to reduce the oxides of nitrogen. The nitrogen was estimated by a method which was essentially that of Dumas. The tellurium dioxide was determined by heating the nitrate slowly until all of the oxides of nitrogen were given off, and then fusing quickly the oxide left. The decomposition of the nitrate was accomplished most conveniently by placing the platinum crucible inside of a larger porcelain crucible, which was heated by a Bunsen burner. After an hour's heating the oxide was in the form of a loose white powder. The results of the analyses follow:—

- I. 0.7975 grm. substance gave 23.25 c.c. N at 0° and 760 m.m. pressure.
 II. 1.2222 grms. substance gave 37.20 c.c. N at 0° and 760 m.m. pressure.
 I. 2.0530 grms. substance gave 0.0538 grm. H₂O.
 II. 2.2981 grms. substance gave 0.0590 grm. H₂O.
 I. 0.9491 grm. substance gave 0.7925 grm. TeO₂.
 II. 0.9920 grm. substance gave 0.8282 grm. TeO₂.

	Calculated for 4TeO ₂ .N ₂ O ₅ .14H ₂ O.	Calculated for 4TeO ₂ .N ₂ O ₅ .H ₂ O.	Found.	
			I.	II.
N	3.62	3.66	3.64	3.84
H ₂ O	3.49	2.36	2.62	2.57
TeO ₂	82.54	83.52	83.49	83.36

The atomic weight of tellurium has been taken as 127.6 in calculating the above results. Klein and Morel (*Bull. Soc. Chim.*, [2], xliii., 198), using 129 as the atomic weight of tellurium, obtained these results:—

	Calculated for 4TeO ₂ .N ₂ O ₅ .14H ₂ O.	Found.	
		I.	II.
N	3.59	3.70	—
H ₂ O	3.47	2.90	3.80
TeO ₂	82.66	82.20	83.30
Te	65.57	66.10	66.50

Experiments described below show that there is no water of crystallisation in the compound. The formula therefore is best written Te₂O₃(OH)NO₃.

Decomposition of Basic Tellurium Nitrate by Heat.

Two samples of the nitrate were heated at gradually increasing temperatures in order to determine whether the hydrogen in the compound is present as water of crystallisation. The substance was heated for two or more hours at intervals of 10°, beginning at 110°. If at the end of that time the weight had changed, the heating was continued without changing the temperature until constant weight was obtained. The results follow:—

Number of hours heated.	Temperature.	Percentage loss.	
		I.	II.
12	110—170°	0.00	0.00
2	170—180	0.07	0.03
4	180—190	0.42	0.26
11	190—200	2.47	3.36
9	200—210	3.71	4.15
9	210—220	4.02	5.85
6	220—230	8.00	9.67
9	230—250	8.60	10.30
1	About 350	16.51	16.50

From the above results it will be seen that there is no loss of weight up to 170°, when a slight decomposition begins. At 190° oxides of nitrogen begin to be evolved. There was no distinct point at which the water was given off. The decomposition was gradual, both water and nitric acid being given off at the same time. The figures in the last line of the table give the results of heating two portions of the nitrate in covered platinum crucibles protected by porcelain crucibles. The results are practically the same as those obtained in the very accurate determinations described later, viz., 16.47. The oxide which was left was tested for nitric acid by phenol-sulphonic acid, and only a trace was found. It is therefore unnecessary to fuse the oxide in making a gravimetric estimation of tellurium, if it is heated in the manner described. The loss which accompanies fusion can be done away with, and more accurate results obtained. The experiments recorded above are not in accord with the statement of Klein and Morel that the nitrate begins to decompose at the fusing-point of lead.

Action of Nitric Acid on the Nitrate.

In re-crystallising the nitrate it was observed that at times a residue was left which dissolved with difficulty in

nitric acid. This residue, examined under the microscope, showed well-developed crystals in the form of octahedra, and, when ignited, it did not lose in weight. The nitrate was changed into a mixture of amorphous and crystalline tellurium dioxide. It was therefore important to study the conditions of formation of the oxide in order to be able to get crystals of the nitrate in perfectly pure condition.

Some nitrate was heated with not enough nitric acid (sp. gr. 1.20) to dissolve it. After the acid had boiled twenty-five minutes the residue was examined and found to contain no nitrate. From the hot solution a small amount of white amorphous powder separated. On evaporation of the nitric acid solution well-defined crystals of the nitrate were formed. These were shown by analysis to be free of oxide. The loss in weight on ignition was 16.51 per cent. Theory requires a loss of 16.48 per cent. The same results were obtained when nitric acid of specific gravity 1.25 was used. It will be seen, therefore, that, while the dilute acids decompose the nitrate, any crystals which are formed from it by concentration are pure nitrate. The reasons for the directions given above for the re-crystallisation of the nitrate are now evident. The nitrate is stirred with the acid (sp. gr. 1.25) at about 70° in order to avoid decomposition into the oxide, and saturation of the solvent. Crystals of the nitrate increase markedly in size when heated with concentrated boiling nitric acid, and a large amount crystallises from the oxide on cooling.

Electrolytic Deposition of Tellurium.

Schucht (*Fakresbericht*, 1883, 222, 1514) and Whitehead (*Journ. Am. Chem. Soc.*, xvii., 849) have shown that tellurium is deposited by the electric current from an acid or alkaline solution. Numerous attempts were made to get the conditions under which the deposit would be made in such a form that it could be weighed. It was deposited from hydrochloric and nitric acid solutions and from solutions of the alkali tellurites. As tellurium is nearest to antimony in the electrolytic scale, the conditions under which antimony is deposited were applied to tellurium. The precipitate was always in an amorphous flocculent condition. Some tellurium which had been deposited by electrolysis was converted into the nitrate and the loss in weight on ignition determined. This was done to determine whether the electrolysis had effected any decomposition of the element. As the loss in weight was 16.55 per cent, it was concluded that no decomposition had taken place.

(To be continued).

ON THE QUALITATIVE SEPARATION OF NICKEL FROM COBALT BY THE ACTION OF AMMONIUM HYDROXIDE ON THE FERRI- CYANIDES.*

By PHILIP E. BROWNING and JOHN B. HARTWELL.

SOME years ago F. W. Clarke (*American Journal of Science*, xlviii., 67) suggested a method for the separation of nickel from cobalt depending upon the solvent action of ammonium hydroxide upon the precipitated ferricyanides. The method may best be described by quoting from the original article:—"To the slightly acid solution containing the two metals, I first add an excess of ammonium chloride. This causes the cobalt precipitate, which otherwise would run through the filter, to fall in a denser state, and also of a much darker colour, often nearly black. I then add the potassium ferricyanide until the precipitation is complete, and afterwards agitate strongly

* Contributions from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, vol. x., October, 1900.

TABLE I.

	CoSO ₄ ·H ₂ O.	NiSO ₄ ·7H ₂ O.	KAl(SO ₄) ₃ saturated solution.	K ₂ FeC ₂ N ₆ .	NH ₄ OH (concentrated).	NaOH, solid, about the size of a pea.	Result.
	Grm.	Grm.	C.m.s.	Grm.	C.m.s.		
1.	—	0·0100	2	0·5	5	"	Heavy precipitate.
2.	—	0·0050	2	0·5	5	"	" "
3.	—	0·0010	2	0·5	5	"	" "
4.	—	0·0003	2	0·5	5	"	Distinct.
5.	—	0·0001	2	0·5	5	"	Plain.

TABLE II.

1.	0·10	—	2	0·5	5	"	None.
2.	0·10	0·0100	2	0·5	5	"	Heavy.
3.	0·10	0·0050	2	0·5	5	"	Distinct.
4.	0·10	0·0030	2	0·5	5	"	Very faint.
5.	0·05	—	2	0·5	5	"	None.
6.	0·05	0·0100	2	0·5	5	"	Heavy.
7.	0·05	0·0050	2	0·5	5	"	Distinct.
8.	0·05	0·0030	2	0·5	5	"	Plain.
9.	0·05	0·0010*	2	0·5	5	"	Faint.

* Equivalent to 0·0002 of the metal.

with a considerable excess of ammonia. Upon filtering, all the cobalt remains upon the filter, being recognised by the characteristic colour of the precipitate, and the nickel is readily detected in the filtrate by means of ammonium sulphide. If, upon filtering, the portion at first running through is turbid, it may be disregarded, or returned to the filter, that which filters through subsequently being almost invariably clear."

In making a study of this method we found two serious objections; first, the practical impossibility of obtaining a good filtration from the cobalt ferricyanide, even in the presence of the ammonium chloride, and, second, the large amount of sulphur thrown down when ammonium sulphide was added to the filtrate containing the nickel with the excess of ferricyanide.

Our first attempt was to secure, if possible, a complete separation of the precipitated cobalt ferricyanide and the dissolved nickel by filtration. This we were able to accomplish by the addition of a small amount of a solution of an aluminum salt to the original solution which held back the cobalt, and, as experiment showed, allowed the complete solvent action of the ammonium hydroxide upon the nickel salt. Amounts of nickel as small as 0·0001 grm. were detected, when mixed with the aluminum salt, by precipitating as ferricyanide, extracting with ammonium hydroxide, and testing in the manner to be described.

On turning our attention to a possible improvement in the method for the detection of the nickel, it was found that when the ammoniacal solution of the nickel ferricyanide was treated with strong sodium or potassium hydroxide solution, in the presence of an excess of potassium ferricyanide, a black flocky precipitate formed which gave no test for ferro- or ferricyanide, and gave every indication of being nickelic hydroxide. This reaction we found to afford us a most delicate test for nickel.

The method as modified by us may be described as follows:—Dissolve not more than 0·1 grm. of the salts of the two elements in about 5 c.m.³ of water, add a few drops of a saturated solution of alum, destroy any free mineral acid by neutralising with ammonium hydroxide, and make faintly acid with acetic acid. To this solution add about 0·5 grm. of potassium ferricyanide, and agitate to effect the solution of the ferricyanide and the complete precipitation of the nickel and cobalt salts. Then add about 5 c.m.³ of strong ammonium hydroxide, and filter. To the filtrate, which should have no reddish colour, add a piece of sodium or potassium hydroxide about the size of a pea, and boil. The appearance of a black precipitate, in case of very small amounts of nickel showing first as a dark colouration, indicates nickel.

The above tables give a record of the experimental results. With the precautions indicated, this method may be applied very satisfactorily.

THE ESTIMATION OF MOLYBDENUM IN IRON.

By E. DOHLER.

TEN grms. of steel, or 10 to 20 grms. of pig-iron, containing 1 to 3 per cent of molybdenum, are dissolved in 100 c.c. of nitric acid: this operation is carried out in a porcelain crucible, first on the water-bath, and finally on a sand-bath; the evaporation is continued to dryness, to separate the silica and to drive off the nitric acid, but the temperature must not be raised too high, or the molybdic acid will be volatilised.

The residue is taken up with about 100 c.c. of hydrochloric acid ($d=1\cdot19$): this solution is then heated on the water-bath and the hydrochloric acid evaporated off, adding water from time to time; finally, when all the oxide of iron is dissolved, and the quantity of hydrochloric acid remaining is not more than 10 c.c., more water is added, the solution is filtered, and the filtrate collected in a flask of about 1·5 litres capacity; the residue is carefully washed with warm water. This residue consists principally of silica; in the case of pig-iron it also contains graphite (and tungstic acid in cases of tungsten steel), and is perfectly free from molybdenum, as has been shown by numerous analyses; if it has been thoroughly washed, its proportion of silica and graphite will not be more than 1 per cent. Through the filtrate, the volume of which is about 1 litre, and which is kept at a temperature of about 80°, a good current of sulphuretted hydrogen is passed for about an hour.

The precipitation of molybdenum in the state of sulphide is very difficult to accomplish, but it can generally be done in one operation by following exactly the instructions given above. The precipitate, which consists principally of sulphur and sulphide of molybdenum, is allowed to stand for twelve hours in the liquid saturated with sulphuretted hydrogen, the beaker being well covered with a watch-glass; it is then filtered and washed five or six times with a cold solution of sulphydric acid, containing a little hydrochloric acid; the filtrate is then evaporated down to half its volume, and again saturated with sulphydric acid to make sure that the precipitation is complete. This is generally the case; if the liquid becomes dark-coloured by reason of the formation of sulphide of manganese—it should be remembered that a

trace of this sulphide suffices, like sulphide of lead, to colour the liquid—it must be left to stand for about ten hours, and then treated as above.

The precipitate is then removed from the filter, and extracted with warm sulphide of ammonium, then with warm water. If any precipitate is left, we try whether it is soluble in hydrochloric acid; if so it is sulphide of iron, and may be neglected; if it become brown, that would indicate that it contained sulphide of molybdenum, and that the extraction with sulphide of ammonium was not complete. The filter must be calcined at a moderate heat in a porcelain crucible, with a little double carbonate of soda and potash and sulphur, in the proportion of 2 to 1, and the melted mass taken up with water. Sulphide of ammonium is added to the solution, and the whole heated for two or three hours to dissolve all the molybdenum. The solution is filtered, and the residue on the filter carefully washed; the residue is treated as before. The solution of sulphide of ammonium, which should be quite clear and of a deep yellow colour, on account of the excess of sulphide of ammonium, is brought to boiling-point, and the molybdenum precipitate d by the addition of hydrochloric acid ($d = 1.124$) in excess. The reaction is violent, especially if the solution is warm, on account of the evolution of sulphydric acid; therefore the hydrochloric acid should be added slowly and cautiously. The solution must then be heated until sulphuretted hydrogen is no longer given off, filtered on a filter dried at 120° , and washed until the filtrate no longer gives any reaction with nitrate of silver. The precipitate is then dried at 120° until its weight is constant.

After this weighing the precipitate is pulverised in an agate mortar. A part of the powder obtained is gently heated to redness in a current of hydrogen until its weight is constant; the pure sulphide obtained (MoS_2 , containing 60.00 per cent Mo) is then weighed.

Although this method is rather complicated, it has the advantage of giving excellent results, which is not the case, as the author has proved, with those methods given in most text-books, and which are, further, almost impracticable.—*Chemiker Zeitung*, 1900, p. 537.

NOTE ON A METHOD OF DETECTING FREE PHOSPHORUS.

By P. MUKERJI, B.Sc., Professor of Chemistry, Presidency College, Calcutta.

THE following statement is made in Roscoe's "Treatise on Chemistry" and in Watts' "Dictionary of Chemistry":—Phosphorus does not directly combine with hydrogen.

The principle employed for the detection of free phosphorus in the present note is the phosphorescence of the vapour of phosphorus diluted with hydrogen gas.

The following is the apparatus employed:—A three-necked Woulfe's bottle; to the middle neck of which there is attached by a cork a tube about 11 inches long and of half an inch diameter, the upper end of the tube being closed with a cork. To another neck a safety funnel with a long tube is attached, and in the third neck a short jet is inserted. The tube of the safety-funnel dips into the liquid in the bottle; the middle tube and the jet enter the bottle only for a short distance. The tube attached to the middle neck is somewhat loosely fitted, so that traces of air may enter the bottle. The jet and the funnel are fitted tightly. The capacity of the bottle may be a litre or less.

For the above-mentioned apparatus a simpler one has been substituted in several experiments; namely, a small flask of about 184 c.c. capacity, and fitted with a funnel with a stopcock and also with a jet.

The Method of carrying out the Test, and the Appearance.—Zinc and dilute sulphuric acid are introduced into the bottle, and the gas issuing from the jet is observed. If in

a dark room, there is no glow perceived, then the materials employed are free from uncombined phosphorus. When the bottle is so hot, through the chemical action between the zinc and acid, that it can scarcely be touched, *i.e.*, when the temperature of the liquid is about 60° to 70° C., and that of the gas in the upper part of the bottle is 45° to 50° C., the cork is removed from the top of the tube attached to the middle neck, and the substance suspected to be phosphorus, or to contain this in a free state, is introduced through the tube into the bottle, and then the cork is quickly inserted. If an organic mixture suspected to contain free phosphorus has to be introduced, it may be poured in through the thistle funnel, or, better still, through the neck to which the jet is attached; the jet is quickly put back into the neck after introduction of the mixture. Soon after the introduction of phosphorus (free) the gas issuing from the jet is found to glow in a dark room, a sheaf of light emanating from the jet. The liquid inside the bottle glows, and luminous flashes are also seen now and then. If now the cork at the top of the middle tube be removed, the light will sink down through the jet, and the gas escaping at the top of the middle tube will glow. On replacing the cork the glow will reappear at the jet. By alternately removing and replacing this cork, the glow may be made to move downwards and upwards through the jet at the option of the operator. (If the second form of apparatus is used, it is only necessary to take off and replace the stopcock below the funnel, and the glow will appear alternately in the funnel and at the jet).

Fresh quantities of dilute sulphuric acid and platinic chloride may be introduced if the glow shows signs of ceasing.

Delicacy of the Test.—In one experiment, 7 m.grms. of phosphorus were cut into two larger and two smaller pieces; and one of the smaller pieces (about 1.5 m.grms.) was introduced into the second form of apparatus; the appearance noted above was observed. After about half-an-hour the zinc was washed two or three times, and kept in the flask under water. About twenty-four hours later this zinc, without any further addition of phosphorus, again showed the glow at the jet and in the funnel.

In another experiment the first form of apparatus was employed, the capacity of the bottle being a little over a litre. 112 grms. of zinc, 250 c.c. of acid and water, and 30 c.c. of milk, were introduced into the bottle; next, 2 m.grms. of phosphorus were introduced. The jet, the middle tube, and the bottle, all glowed in the way described above. It may be here stated that Fresenius's limit for Mitscherlich's test is 1.5 m.grms. of phosphorus in 150 grms. of mixture; the second experiment shows that the present method is, at least, as delicate as Mitscherlich's.

Remarks on the Process.—The use of nascent hydrogen as a reagent for the detection of phosphorus is now new. Valentin mentions it in his "Qualitative Analysis," but his method is not the same as the present method. He observes the green core of the hydrogen jet after ignition, and he does not use a dark room. Moreover, his apparatus is Marsh's apparatus. His test is not a test of free phosphorus only, but indicates indifferently either phosphorus, phosphide, phosphite, or hypophosphite, since any one of these substances will give a flame with a green core on treatment with nascent hydrogen, *i.e.*, with zinc and dilute sulphuric acid. It need hardly be stated that the method described in this paper is not the Blondlot-Dussard method, since the hydrogen jet is not ignited, and the appearance observed is not the green core of the flame of burning hydrogen. The phenomena observed in the present method are very similar to those described by Crookes in page 489 of his "Select Methods," third edition. Crookes, however, uses a somewhat complicated distilling and condensing apparatus, and employs a lamp for distilling the phosphorus; the lamp again necessitates the use of materials for preventing its light from hindering the observation of the glow. Moreover, in his method,

the glow is *not under control*. The apparatus described above is simple; no lamp is required for heating, and consequently no precaution is necessary as to the reflected light of the lamp being mistaken for the glow of phosphorus. Again, the operator can, by using the present method, make the glow move up and down as often as he likes, and with great ease. Finally, there is no risk of explosion, since hydrogen *continues* to be evolved even when air is sucked in through the jet (by opening the top of the middle tube); air cannot rush in to any large extent. Lastly, phosphorus can not only be detected by this method, but it can also be estimated quantitatively by passing the vapour thus evolved into silver nitrate solution.

After determining the exact method in which the present test is to be carried out, the influence of a good many substances upon the glow described above was tried, and by comparing the results obtained with those stated in connection with Mitscherlich's method by Crookes and others, it appears that the *present method is superior to Mitscherlich's*. This will be seen from what will be stated presently. It may be first of all noted that common bazaar zinc was used; the zinc contained some phosphorus in the combined state (and also some arsenic). Hence in contact with dilute sulphuric acid the zinc evolved a jet of hydrogen, which burnt with a green core on ignition, but which gave no glow without introduction of free phosphorus. It follows that for the *detection* of phosphorus it is not necessary to use pure zinc. It may be suggested that steam could be substituted for nascent hydrogen in the second form of apparatus described above. The objection to steam is that the glow is not as controllable as it is with nascent hydrogen; moreover, there is the risk of explosion after suction of air through the jet by removing the lamp from below the flask and *then re-heating*. The risk of explosion will be distinctly greater in presence of mustard oil, phosphoretted hydrogen, &c., and in presence of iodine, sulphuretted hydrogen, ether, &c.; nascent hydrogen has also a decisive superiority, as will appear later on. Then again, on *boiling* phosphorus in a complex aqueous mixture (when the temperature must be higher than 100° C.) some portion of the element may get oxidised by water, if by nothing else, and cannot then be vaporised.

As already stated some of the substances in presence of which phosphorus was detected by this method were such as are known to interfere with the glow in the older methods. Milk, boiled rice, flour, silica, though not included in this category, were thus tried; the glow was well observed both at the jet and at the top of the middle tube after addition of phosphorus. Sodium or potassium hypophosphite solution freshly prepared and filtered from particles of phosphorus gave no glow, though phosphoretted hydrogen was apparently evolved as the gas at the jet burnt with a green core upon ignition; the glow appeared soon after introduction of a minute piece of phosphorus. Phosphoretted hydrogen therefore does not interfere with the glow in this method. Sodium phosphite also gave no glow, though it evolved phosphoretted hydrogen. Nitrous fumes are said to stop the glow of phosphorus, but it was found that in this method nitrate or nitrite mixed with chloride did not prevent the glow. Mustard oil was tried; it did not interfere with the glow; the jet became blocked by a whitish oily deposit after some time, but there was the usual glow in the funnel (of the second form of apparatus). The gas did not take fire, and there was no explosion. Sulphuretted hydrogen and also iodine are stated to interfere with or stop the glow of phosphorus. In the present method the result obtained in presence of either of these substances may be regarded as satisfactory. In one experiment 10 c.c. of a saturated solution of iodine in potassium iodide were introduced in three portions; the glow was seen as usual, and the issuing gas did not colour starch paper to any appreciable degree. In another experiment about 4½ grms. of powdered ferrous sulphide were introduced in two portions, and about 3 m.grms. of phos-

phorus were used. The glow was observed at the jet and in the funnel; next the jet was changed, and the new jet also gave the glow. Ether, alcohol, and oil of turpentine were also tried. Oil of turpentine stopped the glow altogether, but it was found that so far as the present method is concerned the failure may be easily remedied. A piece of phosphorus about 2 m.grms. was introduced into boiled rice; oil of turpentine, 2½ c.c., was next added, and then a little water, and the mixture shaken. Now the turpentine was removed by decantation after adding some water; the mixture was next quickly washed (by decantation) with alcohol, and finally with water. The mixture probably still contained traces of turpentine oil, but on introduction into the hydrogen-evolving bottle the glow was seen as usual. Next 5 c.c. of alcohol were introduced into the bottle, the glow continued at the jet, and was also observed at the middle tube, though it was a little less bright. As regards either it is as prejudicial to the glow as oil of turpentine; but there is this difference that the glow appears *after a time* on introduction of a fresh piece of phosphorus.

It may be stated here that after the experiment is over the bottle or flask used for the experiment should be filled with water before opening it in air.

It will be seen from what has been stated above that the present method will greatly facilitate the detection of phosphorus in food and in cases of poisoning. The apparatus described is simple, and zinc and sulphuric acid can be procured in any town. The glow is seen on a magnified scale, and there is no possibility of its being mistaken, especially as no lamp is required. Moreover, many substances that hinder the glow of phosphorus in the older methods do not affect the glow as observed by the present method.

I have, lastly, to state that my assistant, Haridas Saha, M.A., has helped me considerably in carrying out these experiments.—*Journal of the Asiatic Society of Bengal*, lxi., Part 2, 1900.

Estimation of the Total Sulphur in Coal.—V. Antony and A. Lucchesi.—The authors have modified Eschka's method, and proceed by heating 1 gm. of the material, 4 grms. of pure MnO_2 , 1 gm. of MnO_4 , and 2 grms. of dry NaCO_3 , in a small platinum crucible. The heat is applied gently at first, and finally increased up to a bright red. The mass is then plunged into 40 to 50 c.c. of water, acidulated with nitric acid, boiled, filtered, and the sulphate finally precipitated with BaCl_2 . The authors give a series of comparative analyses made by their method and by that of Eschka, which shows theirs to be as exact as the latter.—*Gazz. Chim. Ital.*, (1), xxix., p. 181.

The Volumetric Estimation of Sulphuric Acid.—F. Litterscheid and K. Feist.—The authors estimate sulphuric acid by treating sulphates dissolved in water with a solution of BaCl_2 (30.5 grms. of $\text{BaCl}_2 + 2\text{H}_2\text{O}$ rlvv, the normal solution being too concentrated). The excess of chloride is precipitated by carbonate of ammonia. The liquid is then titrated with 1/10th normal hydrochloric acid.—*Arch. d. Pharm.*, ccxxxvii., p. 521.

Asbestos Filters.—O. Lohse.—The author has discontinued the use of glass-wool for filters; he proposes, instead, to use test-tubes fitted with ground stoppers, of which the bottoms are pierced with a number of small holes, and with a slight shoulder formed a few centimetres above them. The bottom of these tubes is covered with a layer of short fibres of asbestos, about 10 to 12 m.m. in thickness, which can be introduced by means of a long pair of tweezers. For suction filtering, the tube is passed through a cork fitting in another tube connected with the conical vessel in which the vacuum is made. The author has also constructed a special kind of desiccator; both these pieces of apparatus are illustrated in the original paper.—*Berichte*, xxxii., p. 2142.

NOTICES OF BOOKS.

Water in its Application to Manufactures. ("L'Eau dans l'Industrie"). By H. DE LA COUZ. Paris: Vve. Ch. Dunod. 1900. Pp. 496.

WATER plays a most important part in many manufacturing processes, and its quality, in a large number of cases, makes all the difference between success and failure. The influence of salts soluble in water ought to be more thoroughly studied by the manufacturer, so that he may be able not only to recognise the evil, but also be able to quickly apply the best means for its elimination.

It was with this end in view that the author undertook the preparation of the present volume, and the ground he covers leaves but little undone.

For the generation of steam, which is one of the widest uses to which water is put, numerous objections can be pointed out to the use of different waters; incrustations and corrosions occur which add greatly to the expense of fuel, seriously affect the steaming power of the boiler, and are a fruitful source of explosions. All these inconveniences can be prevented by a previous examination of the available water supply and the timely application of a suitable remedy.

Besides very full instructions for the analysis of water, a large portion of this work is applied to the influence, based on numerous experiments, of waters of various qualities on widely different manufacturing processes, such as dyeing and calico-printing, bleaching, wool-washing, soap-making, tanning, paper-making, photography, brewing and distilling, sugar refining, &c.

In these various industries, the waste water may often be of such a character as to make the recovery of certain portions of their contents a remunerative operation: this unfortunately is often neglected, and, besides the actual pecuniary loss to the manufacturer, the public at large also suffer from the injury done to health by the needless pollution of water supplies. In fact, such purification of waste waters is often, to his own benefit, forced on the manufacturer by public and sanitary authorities.

This work, dealing in so thorough and efficient manner with the subject, reflects the highest credit on the author for the painstaking manner in which he has brought together the large amount of information bearing on such widely differing branches of the subject.

The book is well printed on good paper, and contains numerous illustrations; it has a comprehensive table of contents, but, as is so often the case with books published in France, there is no Index.

The Air of Rooms, an Examination of the Effect produced on the Air of Rooms by the Use of Gas, Coal, Electric Light, &c., for Heating and Lighting Purposes. By FRANCIS JONES, F.R.S.E., F.C.S. Manchester: Taylor, Garnett, Evans, and Co., Ltd. 1900. Pp. 59.

THE chemists of one hundred years ago were agreed that air consisted of two gases, now called oxygen and nitrogen, and that its composition was remarkably uniform. Cavendish found only slight differences between the air of London and the air of Kensington, sometimes in favour of one and sometimes of the other, "but," he says, "the difference was never more than might proceed from the error of experiment, and by taking a mean of all there did not appear to be any difference between them." These views no longer hold good; we know now that air also contains carbon dioxide, water vapour, ammonia, and other gases, in proportions which, though variable, can nevertheless be accurately determined; in most cases, however, the determination of the quantity of carbon dioxide forms a sufficient indication of the quality of the air. The presence of this gas is due either to the breathing of animals or the burning of coal, wood, gas, or other carbonaceous material, both of which processes remove oxygen from the air and form carbon dioxide.

The estimation of carbon dioxide in air is not at all a difficult matter, and is now generally done by what is known as Pettenkofer's process, and it is also now generally agreed that good air, away from towns, contains about 3 volumes of carbon dioxide per 10,000 of air.

The problem of the air of rooms is, however, a very different one to that of the outer air. Indoors the products of respiration and combustion are not removed rapidly, even by the best systems of ventilation, and in many cases they accumulate to such an extent as to become injurious to health.

For the purpose of thoroughly investigating this matter the author fitted up a small room, which was devoted entirely to this investigation. The air space was 935 cubic feet; it had one fireplace, one window, and a ventilator at the roof opposite the door. This ventilator, 18 inches by 9 inches, was always kept open.

Determinations were made of the amount of carbon dioxide at different times of the day, and also of the amount of moisture in the air.

Eleven sets of experiments were made, numbered A to K, under varying conditions, such as—Set A. Coal-fire in use; room lighted with gas. Set D. Gas-fire in use; room lighted with the electric light. Set H. Gas cooking-stove in use (oven), draughting into chimney. Two Bunsen lamps in use; &c. The tests were made of the air at the level of the table, as it is as a rule, says the author, nearest the position where the air of a room is inhaled. We think the author is mistaken here, as the breathing level in a room is decidedly higher than the table level. Full details of these and other experiments are given, and the results are set out in tables and curves at the end of the pamphlet. The author arrives at a number of conclusions as the result of his investigations, among which may be mentioned "The air of a room, however lighted and heated, is purest at the floor, less pure 3 feet above, and most impure at the ceiling." This is exactly what we should have expected, and in fact may be looked upon almost as a matter of common knowledge. He also finds that a coal fire and the electric light is the best method of heating and lighting a room; gas fires diminish the humidity, and the use of gas cooking-stoves—even when draughting into the chimney—greatly increases the amount of carbon dioxide in the air of a room.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 14, October 1, 1900.

Absorption of Free Oxygen by Normal Urine.—M. Berthelot.—The author examined various urines, and found that they all absorb free oxygen to a greater extent than pure water. Under the conditions of his experiments, as much as 22 c.c. per litre was found to be absorbed. Urine behaves as a reducing liquid. In this respect it is like the greater number of the tissues of the body, with this difference that the other tissues exist before the arterial blood passes through them and take up a portion of the oxygen from this blood, whilst urine, on the contrary, is extracted from the blood itself. The absorption of oxygen observed in this set of experiments is a true chemical phenomenon, and it is not due to the agency of microbes, such as that producing acetic fermentation.

Acidity of Urine.—M. Berthelot.—The acidity of urine is usually measured by means of phthalein, and compared with a certain equivalent weight of sulphuric acid. This method, however, needs some modification. To be accurate this weight ought to be replaced by an equivalent

sum of those acids in urine which are susceptible of being estimated by means of phthalein; but this sum includes several orders of acidity, the acidity of the strong acids, such as hydrochloric and sulphuric, which can be accurately estimated with most common indicators, and the acidity of the feeble acids, such as carbonic acid (in the form of bicarbonates), which can be estimated accurately only with certain indicators. So when examining the spectrum it is necessary to use several indicators and compare the results.

Selenides of Nickel.—M. Fonze-Diacon.—Selenium vapour reads at a red heat on powdered nickel, and gives, according to Little, a crystalline mass apparently formed of crystals belonging to the cubic system. The author prepares a series of nickel selenides, analogous to the corresponding sulphides. He obtains the proto-selenide of nickel in very well defined cubic crystals; also the sesqui-, bi-, and sub-selenides of nickel. He finds that hydrochloric acid, even when concentrated and boiling, attacks all these selenides very slightly. Gaseous hydrochloric acid slowly transforms them, at a high temperature, into nickel chloride. Nitric acid oxidises them, giving the selenites. Chlorine easily displaces the selenium at a high temperature. Heated in a current of oxygen for some time, the green oxide of nickel is produced and selenious anhydride. Reduced with hydrogen at a bright red heat, all these bodies give rise to filiform nickel.

Oxycelluloses of Cotton, Flax, Hemp, and Rush Pulp.—Léo Vignon.—The celluloses extracted from cotton, flax, hemp, and rush pulp give practically the same products on oxidation. The differences, established numerically between the properties of the oxycelluloses obtained in the various cases, are relatively slight, and can be explained either by the conditions of the particular physical state of each textile, or by the condensations of the molecule $(C_6H_{10}O_5)_n$, which are not always identical for the various textiles considered. The author examined these various products with regard to oxidation, formation of osazones, reducing power, and fixation of basic colouring matters.

MEETINGS FOR THE WEEK.

THURSDAY, Nov. 1st.—Chemical, 8. "Dehydrohomocamphoric Acid and its Oxidation Products," by Arthur Lapworth. "Derivatives of Ethyl α -Methyl- β -phenylcyanoglutarate," by W. Carter and W. Trevor Lawrence. "The Nitration of Acetamino- o -phenylacetate (Diacyl- o -aminophenol)"—a Correction, by R. Meldola, F.R.S., and Elkan Wechsler. "Rhamnazin and Rhamnetin," by A. G. Perkin and J. R. Allison. "Luteolin" (Part III.), and "Genistein" (Part II.), by A. G. Perkin and L. H. Horsfall. "Colouring-matter of the Flowers of *Delphinium consolida*," by A. G. Perkin and E. J. Wilkinson. "The Action of Alkalies on the Nitro-compounds of the Paraffin Series" (Part II.), by Wyndham R. Dunstan, F.R.S., and Ernest Goulding, B.Sc. "Hexachlorides of Benzonitrile, Benzamide, and Benzoic Acid," by F. E. Matthews. "The Influence of Solvents on the Rotation of Optically Active Compounds" (Part I.), by T. S. Patterson. "Note on Galline's Amidomethylnaphthimidazole," by R. Meldola, F.R.S., and F. H. Streetfield.

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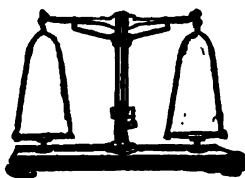
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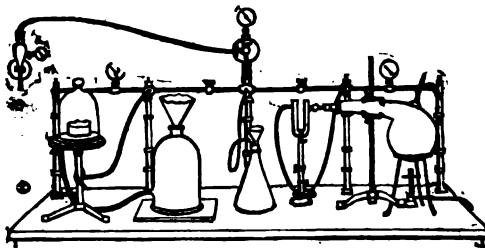
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2136.

KRYPTON II.*

By Prof. A. LADENBURG and Dr. C. KRÜGEL.

IN continuation of our recent communication on this rare element, we desire to state that we have not succeeded in finding any better or shorter method of preparing krypton.

If, as was described in the previous communication, 3 litres of liquid air be allowed to stand for some days in a silvered Dewar flask, and then decanted from the semi-solid paste which separates, the gas collected on allowing the residue to evaporate contains very little carbon dioxide, and, as a closer investigation showed, in addition only oxygen (about 70 per cent), nitrogen (about 28—29 per cent), and argon (about 1 per cent).

After drying, this gas was freed from oxygen and nitrogen by repeatedly leading it over red-hot magnesium previously freed from hydrogen and nitrogen, and then over heated copper oxide. In order to remove the last traces of nitrogen it was mixed, as before, with oxygen, and sparked over potash. When freed from oxygen, dried and collected over mercury, about 1 per cent of the original volume of gas remained. This was condensed by means of liquid air, and gave a clear liquid *without any trace of crystals*. This boiled from about -181.2° to -174° , and was thus almost pure argon, containing at most only traces of krypton.

A similar result was obtained on investigating the residue from the Dewar flask fitted to Linde's apparatus. Here, also, on condensation, an entirely liquid product was obtained, boiling below -170° .

From this it may be concluded that the whole of the krypton of the atmosphere is contained in solution in liquid air, and that the latter does not contain much more than we have isolated from it. Since we began with 850 litres of liquid air, and, according to our earlier determinations (*Ber.*, xxii., 1415), 1 litre of liquid air weighs about 1 kilogram, the amount of material used was about 850,000 grms. From this we obtained about 32 c.c. of krypton, i.e., 0.083 grm., which amounts to only 0.000001 or 0.00001 per cent of the material used. If it be assumed, which is not probable, that half the krypton was lost in working, the upper limit of the proportion of krypton in the atmosphere is 0.00002 per cent.

When the great scarcity of krypton had been established by this research, and there was no prospect of obtaining larger quantities of the substance, it appeared to us to be important to still further test the uniformity of our product, which had been reduced by continued sparking and in preparing a large number of spectrum-tubes to 23 c.c. For this purpose a further fractionation appeared to be the best method. The gas was therefore again condensed by means of liquid air. The condensation-tube became coated with a crystalline layer, but no liquid was produced. By decreasing the pressure and increasing the temperature, the solid was then made to evaporate, and the product was collected in two gasometers, about a third being collected in the first and the remainder in the second vessel. A density determination was then made of the gas in the second vessel.

The following data were obtained:—Weight of gas, 0.0183 grm.; volume of bulb, 7.55 c.c.; temperature, 18° C.; reduced barometer pressure, 745.4 m.m. The density relatively to oxygen = 32, or the molecular weight,

calculated from these data, is 59.01, a result which is in agreement with the numbers 58.81 and 58.67, previously found.

We are therefore justified in concluding that our gas is free from nitrogen and argon, or contains only traces of these, and our hypothesis as to the position of krypton in the periodic system is undoubtedly confirmed by this research. Probably our gas may contain xenon, but the observed density of krypton would then be too high.

THE ESTIMATION OF MANGANESE AND CHROMIUM IN TUNGSTEN ALLOYS.

By FRED. IBBOTSON and HARRY BREARLEY.

THE estimation of manganese by oxidising the nitro-sulphuric solution of the sample with lead peroxide and titrating the permanganate formed, is rapid and accurate for the materials to which it is usually applied. It could be applied to the estimation of manganese in metallic tungsten powders and such tungsten alloys as Fe-W and W-Ni, if it were not that these materials are only partially soluble in the acids usually employed.

By pouring about 10 c.c. of hydrofluoric acid over 1 grm. of the powdered tungsten or tungsten alloy in a platinum basin, adding a few c.c. HNO_3 , and when the reaction slackens 2 or 3 c.c. H_2SO_4 , it goes into solution very easily, and may then be transferred to a beaker, oxidised and titrated with FeSO_4 and KMnO_4 as usual.

The results by this process could be trustworthy only in case the hydrofluoric acid did not react with the permanganate formed; that it does not so react to the detriment of the process was proved by adding 5 and 10 c.c. HF respectively to the solution of a steel containing exactly 0.50 per cent of manganese. The results were 0.495 and 0.504 per cent Mn. And again, to the solution of 1 grm. of W powder obtained in the above manner was added varying amounts of standard manganous sulphate solution. The results calculated to percentages were—

Added.	Found.
0.09	0.08
0.19	0.19
0.38	0.36
0.47	0.46
0.56	0.54

Both tungsten powder and tungsten alloys contain generally only such manganese as is incident to the process of manufacture, and it is less than a half per cent in amount. The glass vessels are hardly attacked by the diluted hydrofluoric acid.

This process cannot be applied to those self hard alloys which occasionally contain as much as 10 per cent of manganese, except by working on less than a decigram. of the material. The chlorate process can, however, be applied if the HF be first boiled off; but it is necessary to remember that the chlorate process, as commonly carried out, gives low results with high manganese alloys, on account of the precipitate containing less oxygen than corresponds to MnO_2 . Both Norris (*CHEM. NEWS*, lxi., 242) and Ford and Bregowkey (*Journal Chem. Soc.*, lxxiv., ii., 540) have used hydrofluoric acid to assist the decomposition of silicious bodies: the latter maintain that the presence of HF assists the precipitation of the manganese.

The Estimation of Chromium.

The alloys of iron, chromium, tungsten, and manganese, which are melted up with iron to produce self-hard steels, are not decomposed by sulphuric or nitric acid, or a mixture of the two, as alloys usually are to which the acid permanganate oxidation, commonly known as Galbraith's process, or Stead's modification of it, are applied,

* Communicated by Prof. van 't Hoff to the Akademie der Wissenschaften zu Berlin, July 5, 1900. Compare *CHEMICAL NEWS*, May 4, 1900, p. 205.

Such alloys may be easily decomposed by acid mixtures containing hydrofluoric, but it is desirable—though not necessary in some cases—to get rid of the hydrofluoric acid again before proceeding to the permanganate oxidation.

Generally the presence of hydrofluoric acid is objectionable, because—unless a large amount of manganous salt is in solution—small additions of permanganate are not decolourised by vigorous boiling when only a small fraction of the chromic oxide has been converted to chromic acid; and, if by the presence of much MnO in solution the permanganate does become all decomposed, the supernatant solution has a clear yellow colour only when a large MnO_2 precipitate has been formed. If a small precipitate only is formed, the filtered solution has a brown colour, due to dissolved MnO_2 or some oxide of manganese higher than MnO . Since the addition of so much permanganate as forms large precipitate carries down some of the chromic acid, Galbraith's process is not trustworthy under these circumstances. But Stead's modification of it, in which the precipitated MnO_2 , purposely a large one, is dissolved in dilute hydrochloric acid and the chlorine boiled off, gives quite satisfactory results when no nitric acid is present in solution.

In order to avoid the use of nitric acid, the sample, after digesting with a mixture of sulphuric and hydrofluoric acids, is treated with a few grms. of permanganate crystals: in moderately dilute solutions the reaction is very rapid, but it is essential, unless capacious platinum basins are available, to transfer to a flask, dilute, and boil with an excess of permanganate until all the metal is gone.

Another method, which is just as expeditious, is to decompose the sample with fluor-nitric acid, add sulphuric, and boil down over a Bunsen until SO_3 is evolved. The sample may then be diluted and completed by either process. A further advantage of this mode of opening up the sample is, that after dissolving up the precipitated MnO_2 with HCl , the tungsten may be estimated by filtering off the WO_3 : the chlorine is boiled from the filtrate all the more quietly for its absence.

The Technical Department,
University College, Sheffield.

THE REDUCING ACTION OF DENITRIFYING BACTERIA.

By G. AMPOLA and C. ULPIANI.

THIS research has been carried out principally on the bacteria which give off nitrogen with the nitrates, especially the *Bacillus denitrificans* V., or *pseudo-pyocyanic*.

Nitrogen combined with hydrogen in the form of ammonia or amine is not set free by the ferment.

Hydroxylamine and hydrazine interfere with the development of the bacteria.

The organic nitrates which are non-saponifiable in aqueous solution, such as nitromethane, remain unattacked.

The saponifiable organic nitrates, such as nitrate of ethyl, are gradually attacked during the saponification.

Among the ionisable nitrates, nitric acid with the ion H is not attacked, even in centinormal solution. The following nitrates are decomposed in the following order, commencing with the one that is the most easily decomposed:—Nitrates of caesium, rubidium, potassium, sodium, lithium, barium, strontium, calcium, magnesium, and glucinum.

The other metallic nitrates are not attacked.

The nitrates of the organic bases, strychnine, cocaine, &c., are rapidly reduced.

The authors propose classifying the denitrifying bacilli in three categories:—

1. Those which destroy nitrates alone.
2. Those which only attack nitrites.
3. Those which act on both nitrates and nitrites.

They have also examined the action of the denitrifying bacilli on arseniates which are reduced to arsenites, on chlorates which give traces of chlorides, and on ferricyanides which are easily reduced to ferro-salts.—*Gazz. Chim. Ital.* [1], vol. xxix, p. 49—72.

NOTE ON M. FITTICA'S TRANSMUTATIONS.

By C. R. GYZANDER,
Chemist to the Cochrane Chemical Co., Boston, U.S.A.

THOUGH I presumed that M. Fittica was sincere when I read his first article about "Transmutation of Phosphorus into Arsenic" (*CHEMICAL NEWS*, vol. lxxxi, p. 257), I nevertheless had the impression that there was a mistake somewhere. When M. Winkler, a little later, showed where the error lay, I was looking forward to Mr. Fittica's acknowledgment of his mistake.

The *CHEMICAL NEWS* of October 5th gave the expected reply from M. Fittica, but, instead of admitting an error on his part, he seems to think that the error is really M. Winkler's. He not only clings to his former statement, but adds that he has succeeded in bringing about another transmutation, that of phosphorus into antimony. M. Fittica, in his first paper, starts from the assumption that his red phosphorus was free from arsenic, "as tested above."

M. Winkler has shown, very conclusively, that those methods, included in "as tested above," are of no value unless properly carried out. Nevertheless M. Fittica, in his second article, says, "Has M. Winkler proved that my methods are inexact or false? In no way." He certainly has in the eyes of most chemists. What is really needed is for M. Fittica to give better proofs that his phosphorus and other reagents are free from arsenic. Though M. Fittica lays great stress on implicitly following his directions, it would have been of inestimable value to him if he had carried out M. Winkler's suggestions in establishing the absence of arsenic.

To publicly state one's belief in, or acceptance of, any hypothesis may be all very proper. To work and search a life-time for facts, by which to prove its correctness, is heroic. One expects learned men to busy themselves with even apparently impossible things, and, to those who do, disappointment and failure should not be surprises. But, on the other hand, one also expects that a man of science should be able to distinguish truth from mere appearance, or, if not, that he should acknowledge the error when it is pointed out to him.

Though I am not in a position to contest the possibility of transmutations generally, or of the doctrine that our elements are compounds, formed by atomic or molecular combinations of one or more truly elementary bodies,—theories which may possess great charm, yet I do know that we are not in possession of any experimental data, including M. Fittica's statements, which might lead us to assume that transmutations are to be transferred to the domain of actual facts.

The reaction which, according to M. Fittica, appears to take place in transmuting phosphorus into arsenic is— $2P + 5NH_4NO_3 = (PN_2O)_2O_3 + 10H_2O + 3N_2$. How does he know that? Why it could not equally well have been $2P + 5NH_4NO_3 = (PN_2O)_2N_3 + 10H_2O + 3NO$, for example, will never be clear to me, unless the latter, or something similar, is reserved for the antimony transmutation. Assuming that it was possible to prepare M. Fittica's hypothetical PN_2O , and that it gave some reactions resembling those of arsenic, it would nevertheless be vastly different from a transmutation. But M. Fittica has not given the slightest evidence to show that the compound obtained by him, and which gave reactions

similar to arsenic, had the composition PN_2O , save the above hypothetical reaction. If the cosmic changes which produced our elements were of such simple nature as indicated in M. Fittica's transmutations, one might assume that the reverse reaction, $\text{As} = \text{Px} + \text{Ny} + \text{Ox}$, was equally readily induced.

If, therefore, M. Fittica can show that arsenic is composed of phosphorus, nitrogen, and oxygen, he will have the means of proving that his assumed reaction is possibly correct, and that a transmutation takes place. Unless M. Fittica can produce better evidence than has been so far submitted, I for one must believe that his conclusions are based on misplaced confidence.

At present the subject may be summed up in the following words:—Unless we do *exactly* as M. Fittica did, and *reason* as he did, we shall probably never be able to realise the truth of transmutations. M. Fittica informs us, in his reply to M. Winkler, that he shall have more to say, later on, concerning the antimony transmutation. Assuming that M. Fittica is equally convinced of the possibilities in this direction, I would venture to suggest that, as the yield of transmuted antimony was so slight, from the phosphorus employed, it might prove advantageous to search amongst the safety matches for reagents to assist in bringing up the yield.

THE RELATIVE VALUES OF THE MITSCHERLICH AND HYDROFLUORIC ACID METHODS FOR THE DETERMINATION OF FERROUS IRON.*

By W. F. HILLEBRAND and H. N. STOKES.

AMONG determinations in mineral and rock analysis, upon the accuracy of which great stress is laid, is that of iron in the ferrous condition. It is of special importance, because an error attaching to it affects in like degree, but opposite direction, the ferric iron that is also nearly always present. Up to about the year 1867, the only method for its determination giving at all satisfactory results was that of A. Mitscherlich (*Journ. Prakt. Chem.*, 1860, lxxxi., 116), depending on the decomposition of the mineral at high temperature by rather strong sulphuric acid (3 parts acid to 1 part water by weight) in a sealed tube from which air has been expelled, and subsequent titration of the ferrous iron by permanganate. When pure hydrofluoric acid became a commercial article, the above method gave place in large measure to one depending on the decomposition of the silicate by a mixture of dilute sulphuric and hydrofluoric acids at atmospheric pressure and a temperature not above 100°C ., care being taken as before to prevent contact with air (*J. P. Cooke, Am. J. Sci.*, 1867, [2], xlv., 347).

It became known in the laboratory of the United States Geological Survey, fully twelve years ago, that the two methods generally give discordant results when applied to the same rocks, the Mitscherlich method in such cases always showing the higher figure for ferrous iron. With low percentages of iron the discrepancies were not very great, but they seemed, in general, to increase as the ferrous oxide rose, until, with a rock showing 10 per cent ferrous oxide by the hydrofluoric acid method, the Mitscherlich method might give 12 per cent. Experiments with artificial ferrous salts throw no light on the subject, for both methods gave with them the same sharp and concordant results.

Until very recently no cause suggested itself whereby the discordance could be explained. In theory, the Mitscherlich method seemed perfect, and it was concluded that, given entire decomposition of all ferruginous minerals,

the determinations made by it were nearer the truth than those by the hydrofluoric acid method. That the reverse is true, in most cases, appears as the result of work done in this laboratory in an entirely different connection.

In the first place it had been found, by long experience, that nearly all rocks carry small amounts of sulphides—pyrite, pyrrhotite, or both—often visible to the eye, but more often in such small amount as to escape direct observation. One of us (Stokes), in an investigation now in progress on the oxidising action of ferric salts on sulphur, pyrite, and other sulphides, has found that this action is vastly more rapid and complete than has hitherto been suspected; that not only is the metal of the sulphide oxidised, but the sulphur to sulphuric acid as well.

The application of these observations to rock analysis shows that the presence of 0.01 per cent of sulphur in any unoxidised form may produce a maximum error of 0.135 per cent in the ferrous oxide determination, and the not infrequent amount of 0.10 per cent of sulphur will multiply this error by 10. One atom of sulphur (32) requires for its complete conversion into trioxide the oxygen of 3 molecules of ferric oxide (480), which then becomes 6 molecules of ferrous oxide (432). The case is still worse with the sulphide, since not only the sulphur, but its accompanying metal must be oxidised. The above percentages of sulphur evolved as hydrogen sulphide, and fully oxidised, would involve errors in the ferrous oxide determination of 0.18 per cent and 1.80 per cent respectively. The error caused by sulphides tends to become greater the more there is present of either or both sulphide or ferric salt. Now the highly ferruginous rocks usually carry more ferric iron than the less ferruginous ones, and they are often relatively rich in pyrite and pyrrhotite; hence the increased discrepancy between the results by the two methods, as the iron contents of the rock rise, is fully in accord with the above explanation.

The following experiments made by Stokes (somewhat out of their contemplated order in his investigation referred to) bear out the above statements most fully, and show that the Mitscherlich method for rocks and all minerals which contain even a trace of free sulphur or sulphides is no longer worthy of a moment's consideration.

Experiment 1.—0.0010 grm. pyrite was heated with 25 c.c. of a sulphuric solution (1 part acid to 3 parts water by weight) of ferric ammonium sulphate (0.0470 grm. of ferric oxide) in a horizontally-lying sealed tube for six hours at 175° to 195°C . All air had been carefully replaced by carbon dioxide. The pyrite had nearly disappeared, and titration with permanganate showed a consumption of oxygen equivalent to 0.0068 grm. ferrous oxide. Complete oxidation of the pyrite would have required oxygen equivalent to 0.0090 grm. ferrous oxide.

Experiment 2.—This was exactly like the first, except that 0.0013 grm. pyrite was used. Permanganate was reduced equivalent to 0.0090 grm. ferrous oxide. Had oxidation been complete the figure would have been 0.0117 grm.

It is quite possible that in both of these experiments the oxidation would have been complete had the pyrite been disseminated throughout a greater space in the tube, as is the case when rocks and minerals are treated, because of the presence of a gm. or thereabouts of diluting quartz and silicates.

Had precisely similar experiments been made with rock powders carrying 0.10 per cent, and 0.13 per cent, of pyrite with 4.7 per cent of ferric oxide and no ferrous oxide, the following erroneous results might have appeared:—

	i.	ii.
Ferrous oxide	0.68	0.91
Ferric oxide	4.00	3.76

To overcome the possible objection that the foregoing experiments were not conclusive as to the worthlessness of the Mitscherlich method in presence of sulphides be-

* Published by permission of the Director of the United States Geological Survey. From the *Journal of the American Chemical Society*, vol. xxii., No. 10.

cause the concentration of the acid was very different from the normal, the following sealed tube experiments were made with acid of the normal strength (3 parts acid to 1 part water by weight) without any ferric salt, the belief being that the stronger acid, under the conditions of the experiment, would itself act as an oxidiser.

Experiment 3.—Pyrite 0.0016 gm. Three hours at 195° C. Solution only partial. Consumption of permanganate equivalent to 0.0017 gm. ferrous oxide.

Experiment 4.—Pyrite 0.0032 gm. Twelve hours at 220° C. Solution perfect. Odour of sulphur dioxide apparent on opening tube. Consumption of permanganate equivalent to 0.01742 gm. ferrous oxide.

Experiment 5.—Sulphur 0.0021 gm. Three hours at 195° C., and, as the sulphur had not disappeared, further six hours at 250° C. Entire disappearance of the sulphur. Consumption of permanganate equivalent to 0.01320 gm. ferrous oxide.

Experiment 6.—Sulphur 0.0039 gm. Twelve hours at 220° C. Entire disappearance of the sulphur, and strong odour of sulphur dioxide on the opening tube. Consumption of permanganate equivalent to 0.03619 gm. ferrous oxide.

In experiments 4, 5, and 6, where decomposition was complete, the consumption of permanganate is considerably less than theory requires, but this is explained by the escape of not a little sulphur dioxide on opening the tube. Had a ferric salt been present the consumption of permanganate would undoubtedly have reached that required by theory.

It might be supposed, from the foregoing, that a similar error would affect ferrous iron determinations by the hydrofluoric acid method. However, experiment has shown that with the amounts of sulphide usually found in igneous rocks, their effect is negligible, though by increasing the amount of sulphide the effect becomes more and more apparent because of the greater surface of pyrite exposed to the action of the ferric iron of the rock.

Under the conditions of the Mitscherlich method, on the other hand—a temperature of 150°–200° C., and even higher, high pressure, much longer time of action, and impossibility of escape of any hydrogen sulphide that may be formed—the sulphur of the sulphides becomes nearly, if not quite, fully oxidised to sulphuric acid, at the expense of the ferric oxide in the rock, with the production of an equivalent amount of ferrous oxide in addition to that resulting from the iron of the sulphide itself.

In order to obtain quantitative data regarding the effect of pyrite on the ferrous iron estimation by the hydrofluoric acid method, the following tests were recently made by one of us.

Part of a fine crystal of pyrite was rather finely powdered and boiled out with dilute sulphuric acid, which extracted considerable ferrous iron, derived presumably from admixed or intergrown pyrrhotite, or from superficial oxidation of the powder, since a second boiling with fresh acid afforded a negative test for ferrous iron. After washing by decantation with water, followed by alcohol and ether, the powder was dried and further pulverised. A quarter of a gm. of it, when treated with dilute sulphuric and hydrofluoric acids in a large crucible by the Cooke method for ferrous iron, then rapidly filtered through a very large perforated platinum cone fitted with filter-paper, required but two drops of a permanganate solution representing only 0.0032 gm. ferrous oxide to the cubic centimetre.

That the error obtaining under the conditions prevailing in rock analysis might be ascertained, successive portions of 1 gm. each of a hornblende schist free from sulphur and carrying 10.09 per cent ferrous oxide as the mean of several determinations and 4 per cent ferric oxide, were mixed in a 60 c.c. platinum crucible with 0.02, 0.025, and 0.10 gm., respectively, of the above purified pyrite powder. This mixture was then treated with hydrofluoric and sulphuric acids as above, the water-bath being at boiling heat for one hour. The cooled contents of the crucible were poured into a platinum dish containing water, and titrated

rapidly nearly to an end. Then, in order to get rid of the pyrite, which would obscure the end-reaction by its reducing effect upon the permanganate, the solution was filtered as above, and in the clear filtrate the titration was carried to completion. The results were 10.02, 10.16, and 10.70 per cent ferrous oxide. Inasmuch as the smallest of these three charges of pyrite was several times greater than what may be considered an unusually high amount for an igneous rock, it is very evident that for all practical purposes the influence of pyrite on the ferrous iron estimation by the Cooke method is negligible. At the same time it is to be borne in mind that with increased content, either of ferric iron or of pyrite, an increased amount of pyrite will be attacked, and that the extent of this attack is undoubtedly influenced by the degree of fineness of the pyrite powder.

All users of the method have noticed the rapid disappearance in hydrofluoric solutions, when titrating ferrous iron, of the pink colour produced by an excess of permanganate. If much ferrous iron be present, many cubic centimetres of permanganate can be added without causing more than a transitory pink coloration. The solution takes on, however, in ever-increasing intensity, the red-brown colour characteristic of manganic salts. It seems that manganous fluoride in acid solution is very susceptible to oxidation by permanganate, for the above-mentioned changes take place when permanganate is added to sulphuric and hydrofluoric acids containing some manganous sulphate.

NOTES ON LECTURE EXPERIMENTS TO ILLUSTRATE EQUILIBRIUM AND DISSOCIATION.*

By JULIUS STIEGLITZ.

I. EQUILIBRIUM AND GASEOUS DISSOCIATION.

THE prominent rôle played by conditions of ionic equilibrium in solutions of electrolytes makes a thorough presentation of the subject desirable in college courses on general chemistry, and a still more exhaustive study necessary in analytical chemistry. The relative size of the dissociation constants, and the influence exerted on the condition of equilibrium by increasing the concentration of one of the dissociation-products, are the points most emphasised. It seems desirable to impress these two points on the mind of the student at the stage where the general subject of equilibrium and dissociation is first dealt with, which may, perhaps, be done best in connection with the question of gaseous dissociation, raised, for instance, by the vapour-density determinations of phosphorus pentachloride, ammonium chloride, &c. The following two lecture experiments, with phosphorus pentabromide and phosphorus trichloridibromide,† were developed to illustrate the second of the two points mentioned, the effect of an increase of the concentration of one of the products of gaseous dissociation. The contrast of the tubes showing the dissociation of the pentabromide and the trichloridibromide at 50° may also serve to demonstrate the first point, the influence of the relative size of the dissociation constant or the relative ease of dissociation, in default of substances for which the constants for gaseous dissociation have actually been determined, and which could at the same time be used for lecture experiments in general chemistry.

Phosphorus Pentabromide and Phosphorus Tribromide.

The effect of an excess of phosphorus tribromide on the dissociation of the pentabromide is shown by comparing

* Contribution from the Kent Chemical Laboratory of the University of Chicago. From the *American Chemical Journal*, vol. xxiii., No. 5.

† Vide Wurtz's work on phosphorus pentachloride, *Ber. Deutsch. Chem. Ges.*, iii., 572.

the intensity of the colour of the bromine vapour in two tubes charged as follows*—

Sealed bulbs containing 0.029 grm. bromine (1 molecule) and 0.058 grm. phosphorus tribromide (a little more than 1 molecule) were placed in a piece of thick-walled tubing† closed at one end, the tube drawn out to a capillary, the air exhausted to 30 m.m. pressure, and the capillary sealed rather close to the tube, the rest of the capillary being bent into a loop. The tube charged in that way was 18 c.m. long, and had a capacity of 40 c.c. By vigorous shaking the bulbs containing the bromine and tribromide were broken. A second tube of the same size was charged with 0.029 grm. bromine (1 molecule) and 0.45 grm. tribromide (9 molecules). A few coils of lead or fuse wire were wound around the lower part of the tubes.

The tubes, thus prepared, are suspended side by side in a tall beaker of water by means of the glass loops at the upper ends. A glazed white porcelain tile placed in a slightly slanting position behind the beaker makes the comparison of the colours easier. On heating at 50° only a very slight colour appears in the first tube (see below, phosphorus trichloridibromide), none in the second tube. From 80° to 90° the dissociation has progressed to the most favourable stage for comparison. The first tube shows a more intense colour than the second one, which contains the excess of the tribromide, the colour of the former being reddish-brown and opaque, while that of the latter is reddish-yellow, through which the white of the tile can still be seen.

Phosphorus Trichloridibromide and Phosphorus Trichloride.

The difference in colour between the two tubes in the above experiment will be sufficiently evident to most students, but perhaps not quite marked enough for the student whose judgment is still to be developed. By using phosphorus trichloridibromide‡ with and without an excess of the trichloride greater differences in colour were obtained, putting the student in question out of temptation to rely less on his own judgment than on the demands of theory.

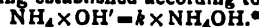
Tubes of the same size (40 c.c. capacity) were charged as above, respectively with 0.029 grm. bromine (1 molecule) and 0.029 grm. phosphorus trichloride (0.004 grm. more than 1 molecule), and with 0.029 grm. bromine (1 molecule) and 0.155 grm. phosphorus trichloride (6 molecules); the air pressure in the tubes was reduced to 27 m.m. At 40°–55° the difference in the colours of the two tubes is most marked, the first one being dark brown and the second one yellow.

The influence of the relative size of the dissociation constants, or the relative ease of dissociation of phosphorus pentabromide and of phosphorus trichloridibromide, is shown by comparing, at 50°, the colours of the tubes containing these substances (the first tube in each series); the former is found to be nearly colourless, while the latter is dark brown, showing the greater tendency of the trichloridibromide to dissociate.

II. EQUILIBRIUM AND ELECTROLYTIC DISSOCIATION. AMMONIUM HYDROXIDE AND SOLUTIONS OF AMMONIUM SALTS (AMMONIUM IONS).

Lövén (*Ztschr. Anorg. Chem.*, xi., 404) has shown that the characteristic action of ammonium salts in preventing the precipitation of magnesium hydroxide by ammonium

hydroxide is simply the result of equilibrium changes. The most important of these is the gradual suppression of the hydroxyl ions of the ammonium hydroxide by greatly increasing the concentration of the ammonium ions on adding the easily dissociating ammonium salts, equilibrium being established according to—



This important action of ammonium salts can be demonstrated very simply by the following experiment† with phenolphthalein. After producing a brilliant red colour by adding a drop or two of dilute ammonia to each of two beakers containing water and a little phenolphthalein, a few drops of a rather concentrated solution of ammonium chloride are added to the contents of one of the beakers. The colour fades to a scarcely perceptible pink, and then disappears.

As ammonium chloride is liable to have an acid reaction, ammonia may be added to its concentrated solution until a little of it can be shown to give a faint but distinct pink colour to a solution of phenolphthalein—proving that it certainly contains no free acid. On adding a few drops of this somewhat alkaline ammonium chloride solution to the crimson solution produced by a drop of ammonia, the red colour instantly gives way to a faint pink. This form of the experiment seems to be the most convincing and striking one; the adoption of either form must depend on the advancement of the class.

It may be added that ammonium chloride solutions which react distinctly alkaline to litmus (see below) make the red colour produced by the action of ammonia on phenolphthalein disappear completely. These experiments are, of course, based on the well-known lack of sensitiveness of phenolphthalein towards hydroxyl ions.

With analytical students the experiment just described may well be followed by a parallel experiment with litmus solution; it is best to use four beakers containing neutral litmus solution; the first is used to preserve the original tint; to the second 5 to 10 c.c. of a concentrated ammonium chloride solution are added, which, to avoid the suspicion of acidity, has been made very slightly alkaline; the colour in this second beaker changes distinctly towards a bluer violet. The other two beakers are each treated with a drop of quite dilute ammonia, which gives to each a pure blue tint, and to one of them 5–10 c.c. of the solution of ammonium chloride are added, the colour reverting to violet. The change is perfectly plain; but, as litmus is more sensitive to hydroxyl ions than phenolphthalein, the change is not as pronounced as when the latter is used.

Küster (*Ztschr. Elektrochem.*, iv., 110) has described a lecture experiment to show, by means of methyl orange, the analogous suppression of the hydrogen ions of acetic acid by adding acetate ions in the form of an acetate (e.g., sodium acetate), according to—



In consequence of hydrolysis sodium acetate reacts alkaline, and the experiment in its original form is, perhaps, open to this objection. A convincing and striking proof of its correctness may be given by using a solution of sodium acetate which, to prevent the suspicion of alkalinity, has been made very slightly acid, giving with methyl orange the well-known reddish-brown hue of beginning acidity. On adding even two or three drops of this slightly acid solution to a beaker of a methyl orange solution, coloured an intense red by a drop or two of acetic acid, the red colour is at once replaced by the brown hue of lesser acidity.

* At the same time $\text{NH}_4\text{OH} = k' \times \text{NH}_3 \times \text{H}_2\text{O}$; the symbols are used to designate concentrations.

† The experiment may serve as a particularly simple and pretty illustration of ionic equilibrium in general, and as a basis for the discussion of the use of ammonium salts with ammonia in many important reactions of analytical chemistry, e.g., in preventing the precipitation of magnesium hydroxide and many analogous hydroxides, in facilitating the quantitative precipitation of aluminium hydroxide, &c. The simpler form is preferable for general chemistry classes, the use of the slightly alkaline ammonium chloride is more instructive for advanced students.

* Instead of using phosphorus pentabromide it was found more convenient to use the tribromide and bromine in molecular proportions, weighed in small bulbs blown at the end of capillaries. The weighing out of the exact quantities required was rapidly accomplished by weighing a bulb both on an analytical balance and on rougher scales. When by means of the latter the amount required in the bulb was very nearly adjusted, the final weighings were made on the sensitive balance, the bulb being placed in a pair of weighing tubes fitting closely over each other.

† The ordinary tubing used for heating solutions under pressure.

‡ Phosphorus trichloridibromide dissociates at 35° into phosphorus trichloride and bromine (Michaelis, *Ber. d. Chem. Ges.*, v., 9).

THE PREPARATION OF PURE TELLURIUM.*

By JAMES F. NORRIS, HENRY FAY, and D. W. EDGERLY.

(Concluded from p. 203).

Fractional Crystallisation of Potassium Bromotellurate.

Owing to the uncertainty of the homogeneity of tellurium, it was subjected to a careful fractionation. Wills (*Journ. Chem. Soc.*, xxv., 704) fused tellurium with potassium cyanide and precipitated the element in two fractions from the aqueous solution of the telluride by a current of air. Brauner used this same process, but separated the tellurium into four fractions. He also precipitated tellurous acid in eight portions from a solution of tellurium tetrachloride, and subjected tellurium tetrabromide to fractional sublimation in a vacuum. The details of the latter method are not given.

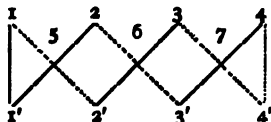
Staudenmaier (*Zeit. Anorg. Chem.*, x., 189) crystallised telluric acid and collected the compound in four portions. In all cases atomic weight determinations of the fractions gave no evidence of a breaking down of the tellurium.

The importance of testing the hypothesis of the compound nature of tellurium led us to undertake a more careful fractionation than had been accomplished heretofore. The results of Wills, Brauner, and Staudenmaier are not conclusive, since the fractionation was not carried far enough in any case to warrant definite conclusions. Crystallisation was selected as the means of fractionation, as it appears to be much more efficient than precipitation. A number of compounds were studied with the view of using them for this purpose. The organic compounds formed by tellurium tetrachloride with anisole and phenetol, $\text{TeCl}_4(\text{C}_6\text{H}_4\text{OCH}_3)_2$, $\text{TeCl}_4(\text{C}_6\text{H}_4\text{OC}_2\text{H}_5)_2$, were prepared. They are described as crystallising well. The progress of the crystallisation could be easily watched, as the compounds have distinct melting-points. It was found extremely difficult, however, to prepare these compounds in large quantities and to re-crystallise them.

The salt finally selected was the double bromide of tellurium and potassium. This salt is readily prepared, and crystallises well from water. In order to have the results of the fractionation as conclusive as possible, all the reagents used were prepared with the greatest care.

Potassium bromide was prepared by heating chemically pure potassium bromate of commerce which had been re-crystallised four times. Hydrobromic acid was made by Squibb's method, and purified by four re-distillations from potassium bromide. The tellurium dioxide was made from basic nitrate which had been re-crystallised three times from nitric acid. The nitrate was decomposed at about 350° , and then fused in portions of 6 to 10 grms. in a platinum crucible protected by a larger porcelain crucible. Theoretical quantities of tellurium dioxide and potassium bromide were dissolved in hydrobromic acid, and the salt obtained by crystallisation. Throughout the work porcelain dishes alone were used. 800 grms. of this compound were then subjected to fractional crystallisation.

The following diagram may help to make the scheme of fractionation clear. Crystals are represented by solid lines and mother-liquors by dotted lines:—



Four portions of 200 grms. each were dissolved in water, usually containing a small amount of hydrobromic acid, and the solutions evaporated to such a point that about 100 grms. of salt separated on cooling. The salt from Fraction 1 was set aside; the salt from Fraction 2 was added to the mother-liquor of Fraction 1, making number 5; the salt from number 3 was added to the mother-liquor

from number 2; and the salt from 4 was added to mother-liquor number 3. The mother-liquor from Fraction 4 was set aside during the intermediate crystallisation of Fractions 5, 6, and 7. These solutions were evaporated as before. The salt from Fractions 1 and 5 were united, making the first portion of the second series. The mother-liquor of number 5 and the crystals from 6 made number 2 of the second series. The mother-liquor of 6 and the crystals from 7 made number 3; and, finally, the mother-liquors of 4 and 7 made number 4 of the second series. It will be seen that a complete series involves seven crystallisations. This procedure of adding the crystals of one fraction to the mother-liquor of another tends to make the most soluble and the least soluble portions collect in the end fractions. The fractionation was repeated thirty times, which involved 215 crystallisations.

There were no indications during the work of any change in the salts, so an atomic weight determination of the tellurium from the end fractions had to be made. More than fifteen different methods have been studied in attempting to determine the atomic weight of tellurium, and of these but one method—the determination of bromine in the tetrabromide—was found to give concordant results. As the use of this method is not free from objections, and involves a large amount of work, a simpler method was sought. Very concordant results were obtained in the analysis of the basic nitrate when it was changed to the oxide by ignition, and it seemed possible that trustworthy results, which would show any variation in the atomic weight, might be obtained, if the ratio between the nitrate and oxide was determined with great care.

As Klein and Mprel have stated that the nitrate is hygroscopic, experiments were made with a pure sample to test this point. Two portions of the salt were heated for eight hours at 120° in platinum crucibles, transferred to a desiccator, and after cooling, were weighed immediately, a platinum crucible being used as a tare. After standing half an hour and fifteen hours in the balance case, the crucibles were weighed again, with the following results:—

Hours.	A.	B.
0	1.96242	2.19839
0.5	1.96275	2.19860
15.0	1.96274	2.19855

It will be seen that the weights remained perfectly constant after the crucibles had stood long enough to be in equilibrium with the atmosphere of the balance case, which shows conclusively that the salt is not hygroscopic. It was then heated at 120° for varying periods of time to determine whether it could be brought to constant weight.

Hours heated.	A.	B.
2.5	2.28692	2.12905
14.0	2.28667	2.12897
7.5	2.28676	2.12896
7.0	2.28672	2.12905

The above results show that this could be done very satisfactorily.

The preliminary experiments on the decomposition of the nitrate showed that the oxides of nitrogen evolved during heating carried off a small amount of tellurium dioxide mechanically. In order to avoid this loss, the following device was used:—A platinum crucible was provided with two removable platinum discs which fitted the crucible at distances of about one-half and one inch from the bottom. In the centre of the lower disc a hole was cut about one-eighth inch in diameter. The salt was placed in the crucible, the discs adjusted and the cover put on. There were three surfaces over which the issuing gases had to pass, and on these the oxide carried along was deposited. In the experiments a slight dulness was visible on the upper disc, but the cover was bright, thus showing that no oxide had reached it.

The nitrate was decomposed slowly, in order to avoid a rush of gas. It was heated for two hours at 200° , 250° , and 300° ; three hours at 350° , 400° , 450° , 500° , and 550° ;

* Contributions from the Chemical Laboratories of the Massachusetts Institute of Technology. From the *American Chemical Journal*, vol. xxiii., No. 2.

and was finally fused quickly in the oxidising flame of a Bunsen burner.

The tellurium from the two end fractions of the fractional crystallisation was converted into nitrate. The salt was moistened with concentrated nitric acid when being ground, in order to prevent decomposition from atmospheric moisture. The finely-divided nitrate was heated to constant weight and converted into the oxide in the manner described above.

The results of the analysis of the nitrate prepared from the tellurium from the fraction which would contain the most soluble portion, follow:—

	$\text{Te}_2\text{O}_3(\text{OH})\text{NO}_3$	TeO_2	Percentage TeO_2
I.	2'84426	2'37354	83'45
II.	2'55736	2'13397	83'44

The analysis of the nitrate from the fraction which would contain the least soluble portion, was made under the same conditions as before:—

	$\text{Te}_2\text{O}_3(\text{OH})\text{NO}_3$	TeO_2	Percentage TeO_2
III.	1'39948	1'15824	83'48
IV.	1'85244	1'54649	83'49

The difference between the average of determinations I. and II., and the average of III. and IV. corresponds to a difference of 0'4 in the atomic weight. When a sample weighing 2 grms. is used, an error of 0'3 m.grm. in the weight of the nitrate would affect the atomic weight to this extent. Although the weights of the nitrate in the above experiments appear to be accurate to 0'1 m.grm., nevertheless the difference found is probably more apparent than real.

The basic nitrate is a crystalline compound and may have held mother-liquor within the crystals, although the salt was ground to a fine powder and did not lose weight when heated for ten hours at 120°. On account of this uncertainty, we do not consider the results as data which can be used for calculating the atomic weight of tellurium. The results are of value, however. The analyses of the nitrate prepared from the fractional tellurium were made under exactly the same conditions, and any error in the method would have affected the two determinations equally. We can, therefore, safely conclude that the fractionalisation of potassium bromotellurate did not effect any decomposition of the tellurium, which could be detected by a method capable of giving results accurate to 0'4 of a unit in the atomic weight.

The work described above is preliminary to an atomic weight determination which is now in progress. When a method has been devised which will give accurate results, the atomic weight from the end fractions will be determined, in order to decide whether the difference of 0'4 of a unit, indicated by the experiments given above, is a real one or not. We hope also to offer more conclusive evidence in regard to the elementary nature of tellurium. The fractional sublimation of tellurium dioxide, which is now in progress, seems to offer a more efficient means of deciding this point than fractional crystallisation.

This paper has been published in this unfinished form, as the departure of one of us from the Institute prevents us from continuing the work in common.

The Boiling-point of Colloidal Solutions of Salts in Water.—F. Kraft.—The author has examined the retarding action to the boiling of water, of sodic or potassic soaps, palmitates, stearates, erucates, and oleates. In spite of all the precautions taken to prevent overheating (glass beads, &c.), this retardation is excessively slight; for example, only one-tenth the normal retardation, sometimes nothing at all; this arises from the colloidal nature of the salt in solution. If we then add crystalline salts, such as NaCl, we observe the increase of the boiling-point, which the latter should bring about. On the other hand, in solutions of absolute alcohol the soaps behave normally as non-dissociated crystalloids. The presence of a little water considerably increases the calculated molecular weight.—*Berichte*, xxxii., p. 1584.

THE ALLOTROPIC FORMS OF SELENIUM.*†

By A. P. SAUNDERS.

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I. INTRODUCTION.

The present investigation was taken up for the purpose of establishing the general relationships of the allotropic forms of selenium, and of determining their limits of stability. The descriptions commonly given are confusing because no systematic study of the subject has ever been made. The only available data which could lead to any conclusions are those relating to specific gravities, but these have often been determined without sufficient attention being paid to the methods by which the materials were prepared, or the conditions under which accurate results can be expected. Since none of the text-books give any adequate review of the literature dealing with this element, it is proposed to gather together here a few notes on the principal memoirs which have appeared since the discovery of selenium in 1818. This review will be strictly limited to those which deal with the properties of the element itself, neglecting those devoted to its compounds.

For convenience in discussing the earlier memoirs, the conclusions to which the present investigation has led are given below, after the list of references to the literature. Following these, there comes a description of the method of purification used, and then a discussion of the results previously recorded in the literature and those which the present experiments have brought out; these experiments include specific gravity determinations, dilatometric measurements, experiments on the behaviour of selenium in liquids, and miscellaneous observations; here follow also sections on the heat of transformation, specific heat, conductivity, &c. Finally, there will be found a section devoted to a description of the general physical properties, such as the atomic weight, vapour-density, &c., which are only inserted here for the sake of completeness.

* From the *Journal of Physical Chemistry*, vol. iv., No. 6.

† The change to *selenium* advocated in "Watts's Dictionary" because of the pseudo-metallic character of the element seems unnecessary; the statement made there to the effect that Berzelius so named it, is incorrect. Berzelius called it *selenium* while expressly recognising it as pseudo-metallic, between sulphur and tellurium.

II. LIST OF REFERENCES.

NOTE.—It should be noted that I have used the abbreviation "Ann." for both the *Annales de Chimie et de Physique* and *Liebig's Annalen*. Since the series number always accompanies references to the former, no ambiguity is possible.

1818. Berzelius (*Schweigg. J.*, xxiii., 309—344, 430—484).—Discovery, separation, character, compounds, atomic weight.
1826. Seebeck (*Pogg.*, vi., 155).—Electrification of vitreous selenium.
- Berzelius (*Pogg.*, vii., 242).—Purification.
- Berzelius (*Pogg.*, viii., 21).—Atomic weight.
1827. Mitscherlich and Nitzsch (*Pogg.*, ix., 627—630).—Selenic acid, and salts.
- Magnus (*Pogg.*, x., 491).—Solubility in sulphuric acid.
1828. Fischer (*Pogg.*, xii., 153—155).—Solubility in sulphuric acid.
- Magnus (*Pogg.*, xiv., 328).—Solubility in sulphuric acid.
1829. Fischer (*Pogg.*, xvi., 121).—Solubility in sulphuric acid.
1830. Magnus (*Pogg.*, xx., 165—166).—Separation from compounds.
1831. Rose (*Pogg.*, xxi., 431).—Sulphur, selenium, tellurium.
1839. Knox (*Trans. Royal Irish Acad.*, 1843, xix., 149; or *Phil. Mag.*, [3], xvi., 185).—Conductivity.
1840. Regnault (*Ann.*, [2], lxxiii., 51).—Specific heat.
- Fröbel (*Pogg.*, xlix., 590—591).—Crystal form of the metallic modification.
1845. Riess (*Pogg.*, lxiv., 50).—Electrification of vitreous selenium.
1847. Sacc (*Ann.*, [3], xxi., 119—126).—Atomic weight.
1848. Schaffgotsch (*J. Pr. Chem.*, xliii., 308—309).—Specific gravities.
1849. Erdmann and Marchand (*J. Pr. Chem.*, 1852, lv., 202).—Atomic weight.
1851. Hittorf (*Pogg.*, lxxiv., 214—220).—Melting-point of the metallic form, 217°. Transformation from vitreous to metallic causes evolution of heat. Specific gravity of metallic form. Vitreous selenium a non-conductor. Metallic selenium a conductor.
1853. Schaffgotsch (*Pogg.*, xc., 66—82).—Specific gravities.
1855. Mitscherlich (*Berl. Akad.*, 409—416; or *J. Pr. Chem.*, lvi., 257—265; or *Ann.*, [3], xlvii., 301—313).—Description of the red crystalline form, its crystallography, density, melting-point; transformation of amorphous and vitreous selenium in carbon disulphide.
1856. Regnault (*Ann.*, [3], xlvii., 281—301; or *Pogg.*, xcvi., 418—434).—Specific heat, transformation, general properties, heat of transition.
1857. Oppenheim (*J. Pr. Chem.*, lxxi., 279—282).—Separation from tellurium by potassium cyanide.
- Böttger (*J. Pr. Chem.*, lxxi., 512).—Decomposition of selenide solutions.
1859. Dumas (*Ann.*, [3], lv., 186—187).—Atomic weight.
- Deville and Troost (*C. R.*, xlix., 239).—Vapour density.
1860. Uelsmann (*Ann.*, cxvi., 122).—Various compounds.
1863. Deville and Troost (*C. R.*, lvi., 891).—Vapour density.
- Werther (*J. Pr. Chem.*, lxxxviii., 180—181).—Spectrum.
1865. Böttger (*J. Pr. Chem.*, xciv., 439—440).—Separation by dissolving in sodium sulphite solution.
- Neumann (*Pogg.*, cxix., 123).—Specific heat and specific gravity of the metallic form.
- Plücker and Hittorf (*Phil. Trans.*, clv., 1—29).—Spectrum.
1866. Schneider (*Pogg.*, cxviii., 327—334).—Solubility in selenious bromide; preparation of that substance.
1868. Bettendorf and Wüllner (*Pogg.*, cxxxiii., 293).—Specific heat of vitreous and metallic selenium. Specific gravity of the metallic form.
- Quincke (*Pogg.*, cxxxv., 629).—Capillarity constant.
1869. Fizeau (*C. R.*, lxxviii., 1125).—Coefficient of expansion.
- Schultz-Sellack (*Berl. Akad. Ber.*, 745; or *Pogg.*, cxxxix., 182).—Diathermancy.
- Rathke (*J. Pr. Chem.*, cviii., 235—254 and 321—356; or, somewhat condensed, *Ann.*, clii., 181—220).—Analogies between selenium and sulphur. Various selenium compounds.
1871. Sirke (*Pogg.*, cxliii., 429—439).—Refraction and dispersion of amorphous selenium.
- Ditte (*C. R.*, lxxiii., 622).—Spectrum.
1873. Petersson (*B.*, vi., 1466—1477).—Separation by means of potassium cyanide.
- Salet (*Ann.*, [4], xxviii., 47—49).—Spectrum.
- Sale (*Proc. Roy. Soc.*, xxi., 283—285; or *Pogg.*, cli., 333—336).—Conductivity and light effect.
- Smith (*Am. J. Sci.*, [3], v., 301).—Conductivity and light effect.
1874. Rosse (*Phil. Mag.*, [4], xlvii., 161).—Conductivity and light effect.
- Rammelsberg (*Pogg.*, clii., 151—157; or *B.*, vii., 669—670).—Different modifications, crystallography, density.
- Nilson (*B.*, vii., 1719—1721).—Separation by potassium cyanide.
- Hilger (*Ann.*, clxix., 211—212).—Solubility in sulphuric acid.
1875. Siemens (*Pogg.*, clvi., 334—335).—Influence of light on conductivity (preliminary notice).
- Adams (*Proc. Roy. Soc.*, xxiii., 535—539).—Conductivity and light effect.
- Siemens (*Dingl. Pol. J.*, ccxvii., 61—63).—Electrical photometer.
1876. Petersson and Ekman (*B.*, ix., 1210—1212).—Atomic weight.
- Draper and Moss (*CHEM. NEWS*, xxxiii., 1).—Conductivity and light effect.
- Moss (*CHEM. NEWS*, xxxiii., 203).—Effect of mercury on conductivity of selenium.
- Adams (*Proc. Roy. Soc.*, xxiv., 163—164; or *Pogg.*, clix., 629—631).—Conductivity and light effect.
- Siemens (*Pogg.*, clix., 117—141; or *Berl. Akad.*)—Effect of light and heat on the conductivity of selenium.
- Adams and Day (*Proc. Roy. Soc.*, xxv., 113—117).—Conductivity and light effect.
1877. Braun (*Wied.*, i., 95).—Conductivity and light effect.
- Forssmann (*Wied.*, ii., 512—521).—Conductivity and light effect.
- Siemens (*Wied.*, ii., 534).—Conductivity and light effect.
1878. Sabine (*Phil. Mag.*, [5], v., 401—415).—Conductivity; effect of light and heat.
1879. Carnelley and Williams (*CHEM. NEWS*, xxxix., 286).—Boiling-point.
- Cross and Higgin (*J. C. S.*, xxxv., 253).—Action on water.
1880. Breguet (*Ann.*, [5], xxi., 560—563).—Photophone.
- Obach (*Nature*, xxii., 496).—Effect of phosphorescent light on conductivity.
- Bell (*Proc. Amer. Assoc. Adv. Sci.*, 115—136; or *Ann.*, [5], xxi., 399—430).—Photophone.
- Blondlot (*C. R.*, xcii., 882).—Electrical properties.
1881. Kälischer (*Carl's Rep. de Phys.*, xvii., 563—570).—Photophone without battery.
- Moser (*Phil. Mag.*, [5], xii., 212).—Action between selenium and metals, &c.
- Bidwell (*CHEM. NEWS*, xliii., 105).—Telephotograph.
- Mercadier (*C. R.*, xcii., 789 and 1407).—Photophonic receivers.

1881. Spring (*Bull. Acad. Belg.*, [3], ii., 88; or *Cb.*, III., xiii., 146).—Coefficient of expansion.
— Boraträger (*Dingl. Pol. J.*, ccxlii., 55).—Preparation of metallic crystals by sublimation.
1882. Troost (*C. R.*, xciv., 1508–1510).—Boiling-point.
— K'enen (*Bull.*, xxxvii., 440–443).—Method of extraction.
1883. Bidwell (*Phil. Mag.*, [5], xv., 31–35).—Conductivity, and heat and light effects.
— Schuller (*Wied.*, xviii., 319).—Distillation of selenium.
— Fritts (*Am. J. Sci.*, [3], xxvi., 465–472).—Conductivity and light effect.
1884. Hasebus (*Journ. Russ. Phys.-Chem. Soc.*, xv., 123 and 146; or *Carl's Rep. d. Phys.*, xx., 490–499 and 565–577).—Theory of light effect.
— *Brit. Assoc. Rep.*, 440.—Spectrum.
— Bidwell (*CHEM. NEWS*, li., 261 and 310).—On the resistance of sulphur and selenium cells.
— Divers and Shimosé (*CHEM. NEWS*, li., 199).—Separation of selenium and tellurium.
1885. Schulze (*J. Pr. Chem.*, [2], xxxii., 390–407).—Soluble selenium.
— Bidwell (*Phil. Mag.*, [5], xx., 178–191).—Conductivity and light effect.
— Bidwell (*CHEM. NEWS*, lii., 191–193).—Light sensitiveness due to selenium.
— Divers and Shimosé (*B.*, xviii., 1209–1211).—Separation from tellurium by sulphuric acid.
— Morize (*C. R.*, c., 271).—Selenium actinometer.
1886. Fabre (*C. R.*, ciii., 53–55).—Heat of transformation, vitreous into metallic. (See 1887).
1887. Fabre (*Ann.*, [6], x., 472–550).—Heat of transformation.
— Bellati and Lussana (*Gazz. Chim. Ital.*, xvii., 391; *Fahresb.*, 207).—Heat conductivity of metallic selenium increased by light.
— Kalischer (*Wied.*, xxxi., 101–108).—Electromotive force and light effect.
— Muthmann (*B.*, xx., 990).—Soluble selenium.
1888. Righi (*Wied. Beibl.*, xii., 683).—Electromotive force of selenium in the light.
— Uljanin (*Wied.*, xxiv., 241–273).—Electromotive force of selenium in the light.
— Kalischer (*Wied.*, xxxv., 397–399).—On the articles of Righi and Uljanin.
1889. Righi (*Wied.*, xxxvi., 464–465).—Reply to Kalischer.
— Korda (*J. de Phys.*, [2], viii., 231; or *Wied. Beibl.*, xiv., 50).—Conductivity.
1890. Muthmann (*Z. f. Kryst.*, xvii., 336–367).—Preparation and properties of the two monoclinic red forms. Crystallography of these and of metallic selenium.
1891. Petrusen (*Zeit. Phys. Chem.*, viii., 612–616).—Allotropic forms, heat of transition, density.
— Bidwell (*Phil. Mag.*, [5], xxxi., 250–256).—Conductivity and light effect.
— Muthmann (*Zeit. Phys. Chem.*, viii., 396–397).—Isomorphism with sulphur.
— Retgers (*Zeit. Phys. Chem.*, viii., 72).—Isomorphism of sulphur, selenium, tellurium.
1893. Retgers (*Zeit. Anorg. Chem.*, iii., 343).—Solubility in methylene iodide.
1897. Tammann (*Wied.*, lxii., 280).—Two melting-points.
1898. Klages (*Chem. Zeit.*, xlii., 449–450; *Cb.*, II., ii., p. 253).—Decomposition of selenious acid by hydrogen.
— Lenher (*J. Am. C. S.*, xx., 555).—Atomic weight.

III. THE FORMS OF SELENIUM (IN BRIEF).

Selenium exists in three distinct forms:—

1. Liquid (including vitreous, amorphous, and soluble selenium).
2. Crystalline red (including perhaps two closely allied forms).
3. Crystalline grey or metallic.

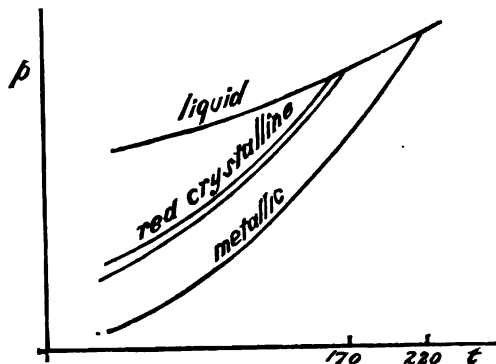
1. Liquid selenium has the properties of an ordinary liquid at temperatures above 220°. Below that it becomes gradually more and more viscous, remains soft down to about 60°, and at 30°–40° gets quite hard and brittle. In this form it is ordinarily known as vitreous selenium, from its conchoidal and brilliant fracture, and it is by that name that it will be called in the following pages. Its streak is red, and it yields on powdering at first a grey powder, but if rubbed up very fine this becomes red, and cannot then be distinguished from the form known as red powdery, or amorphous selenium, which I shall designate by the latter name. Amorphous selenium is the state in which the element separates from solutions of selenious acid on reduction; it forms, when dry, an impalpable powder without any trace of crystallinity. When warmed to 40°–50° it darkens in colour and coagulates to a soft mass, which, when cooled, becomes hard and brittle again, assuming somewhat the fracture of the vitreous form. Amorphous selenium shows all the properties of the vitreous form, making due allowance for the difference in the state of aggregation—the only difference which separates them.

Liquid selenium, whether amorphous or vitreous, is soluble to a slight extent in carbon disulphide.

Amorphous selenium, when freshly precipitated, under certain conditions, from selenious acid solutions, is soluble in water, and in this state it is known as soluble selenium. The property is lost with lapse of time.

2. The red crystalline form separates from solutions in carbon disulphide, or may be obtained by simply allowing the amorphous or vitreous form to stand in contact with that or some other solvent at ordinary temperatures. It is probable that it has two different crystal habits, both belonging to the same system, and both soluble in carbon disulphide; there may be a slight difference of stability between these two; there are indications of an unstable melting-point at about 170°.

3. Grey crystalline or metallic selenium, as I propose to call it, is obtained from any of the above forms at higher temperatures; in presence of certain liquids the change takes place even at ordinary temperatures. The ease with which the transformation can be effected depends upon several factors, but the same form always results. This is the stable form of selenium, at all temperatures from the ordinary temperature to the melting-



point, 217° C. The other two forms are unstable, the red crystalline one representing an intermediate stage between liquid and metallic selenium. It has not proved possible to effect a reverse transformation from the metallic into any other form below 217°, hence there is no real transition point in the physical property curves of the element.

Thus the curves representing the changes in vapour-pressure for changes in temperature should be, in their general character, somewhat as shown in the accompanying drawing (Fig. 1).

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, October 26th, 1900.

Dr. O. J. LODGE, F.R.S., President, in the Chair.

THE CHAIRMAN read a letter from Prof. CLEVELAND ABBE, of the United States Coast and Geodetic Survey, stating that the *Monthly Weather Review* would be sent regularly to any Member of the Physical Society expressing a wish to receive it. On the other hand, the Chief of the Weather Bureau would, at any time, be glad to receive communications referring to the Physics of the Atmosphere.

Dr. SHELFORD BIDWELL then exhibited some "*Experiments illustrating Phenomena of Vision.*" The first phenomenon illustrated was that known as "*Recurrent Vision.*" A vacuum tube, illuminated by an induction coil, was made to rotate about a horizontal axis, and was seen to be followed, at an angle of about forty degrees, by a feebly luminous reproduction of itself. A spot of white light, projected upon a screen, and caused to move slowly in a circular path, was also followed by a less luminous spot. The same effect was shown by spots of green and yellow light, but in the case of red light no ghost was visible. The phenomena of recurrent vision are due principally, if not entirely, to the action of violet nerve fibres. The next experiments related to the non achromatism of the eye. The lenses of the eye do not constitute an achromatic combination, although under ordinary conditions a bright object is not surrounded by fringes of colour. The effects of chromatic aberration are disguised by the luminous haze which surrounds the object, produced by a defect in the eye regarded as an optical instrument. A six-rayed star, formed by cutting a hole in an opaque screen, was illuminated by a gauze-covered condenser containing an incandescent lamp. The star was fairly clearly defined, and there were no fringes. More attentive observation showed a luminous haze. This haze is formed in consequence of the cellular structure of the eye, and the brightest rays—orange, yellow, and green—are chiefly instrumental in forming it. If therefore these rays are obstructed, the conditions are more favourable for the observation of chromatic aberration. The rays were consequently cut off by means of coloured glasses, and the general hue of the star was purple; to some it appeared bordered with dark blue, while to others (long-sighted) it appeared bordered with red.

Two oblong patches, one red and the other blue violet, and of approximately the same intensity, were then produced, side by side, upon a screen. An observer with very good eyesight was able, at a distance of 10 feet, to focus the patches alternately with perfect distinctness. In general, the blue patch was said to be more or less blurred. With an achromatic eye it should be possible to focus both together.

Dr. BIDWELL then showed some lantern-slides, illustrating the complex form seen when viewing a small luminous spot through a gauze-covered lens placed so as not to be in exact focus.

Some experiments were performed illustrating the principle of the colour top. When a bright image is formed on the retina after a period of darkness, it has, in general, a red border, which lasts for a fraction of a second. A dark patch, suddenly formed on a bright ground, has a blue border, which lasts for a similar time. These effects were attributed by Dr. Bidwell to a sympathetic action of the red nerve-fibres. When the various nerve fibres occupying a limited portion of the retina are stimulated by ordinary white or yellow light, the immediately surrounding red nerve-fibres are for a short period excited sympathetically, while the violet or blue and green fibres are not so excited or in a much less degree. Again, when light is suddenly cut off from a patch in a bright field, there occurs a sym-

pathetic insensitive reaction in the red fibres just outside the darkened patch, in virtue of which they cease for a moment to respond to the luminous stimulus; the green and violet fibres, by continuing to respond uninterruptedly, give rise to the sensation of a blue border. By a simple experiment it was shown that the explanation of the colour top, depending upon changes in the convexity of the eye and non-achromatism, was untenable. By the use of a strong light it is possible to get negative after-images, after looking at a brightly coloured object. These images are complimentary in colour to the object, and are formed even if the object is only viewed for a fraction of a second. By means of proper illumination and a disc rotating at the proper speed, a red wafer was so arranged that, upon looking at it, it was impossible to recognise the wafer itself, but only the continuous green after-image.

THE CHAIRMAN expressed his interest in the last experiment, in which it was possible to see the negative after-image of an object and not the object itself.

Prof. S. P. THOMPSON said these experiments threw a doubt on some of the accepted notions about the properties of the eye. Dr. Bidwell asks us to believe that the yellow haze is due to a cellular structure in the eye. Is there such a structure? can it be observed with a microscope? and do its meshes correspond in magnitude with those necessary to produce the effects? By diminishing the size of the pupil the haze is diminished, and the sharpness of the image is increased. The effects seem to be due to ordinary aberration. Prof. Thompson said that the achromatism of the eye was simply shown by covering half the object-glass of a telescope and viewing a bright object with it. The object then seems bordered with coloured fringes.

Mr. BLAKESLEY, referring to the colour patches used by Dr. Bidwell, pointed out that, although the patches were the same distance from the lens, yet they did not possess the same magnification. The last experiment shown did away with the theory of persistence of vision, because the space between the object and the negative after-image was evidently not illuminated.

Mr. TROTTER asked if red and green were the only colours which gave complimentary negative after-images.

Dr. BIDWELL, in reply, said the effect was obtainable throughout the length of the spectrum.

A paper on "*The Concentration at the Electrodes in a Solution, with special reference to the Liberation of Hydrogen by Electrolysis of a Mixture of Copper Sulphate and Sulphuric Acid,*" was read by Dr. H. J. S. SAND.

In this paper an equation has been derived for calculating the concentration at the electrode of a solution of a single salt from which the metal is being deposited under the conditions: (1) that the solution is contained in a cylindrical vessel bounded by the electrodes; (2) that no convection-currents occur; and (3) that the diffusion of the salt obeys Fick's law, and its transport values are constant. This formula can be made the basis for roughly determining diffusion coefficients. In the case of mixtures it is possible to arrive at limits for the concentration, and it has been experimentally proved that hydrogen always appears at the electrode of an acid solution of copper sulphate, in which no currents of liquid are taking place, between the limits of time for the concentration to go down to zero. The time which it takes for the hydrogen to appear can be calculated from an empirical formula, which is similar in form to the one used for a single salt. The great part played by convection-currents in determining the ratio of the two constituents given off at the electrode of an acid copper sulphate solution, has been shown by proving experimentally that artificial stirring causes hydrogen to disappear altogether in cases where it would otherwise have presented over 60 per cent of the equivalents carrying the current from the solution to the electrode.

THE CHAIRMAN drew attention to the fact that no hydrogen was liberated until all the copper had gone, and said

the formula for the concentration might be used again in further investigations.

Dr. DONNAN asked if the time at which hydrogen was liberated had been taken as the time at which hydrogen actually made its appearance in the form of bubbles, or whether any allowance had been made for saturation.

Dr. SAND said the time was taken up to the appearance of bubbles.

A paper by Dr. R. A. LAHFELDT on "*Electromotive Force and Osmotic Pressure*" was postponed until the next meeting.

The meeting then adjourned until November 9th.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxi., No. 15, October 8, 1900.

Iron Silicide, SiFe_2 , and its presence in Industrial Ferro-silicons.—P. Lebeau.—The author prepares crystalline silicide of iron, SiFe_2 , by the action of copper silicide on excess of iron at a high temperature. The fused mass is treated with dilute nitric acid to completely dissolve out the cuprous compounds, when the iron silicide remains. The crystals are brilliant and have an iron-grey colour. This compound is identical with the silicide already described by M. Henri Moissan. It is only slightly attacked by acids and alkalis, with the exception of concentrated or dilute hydrochloric acid, which completely dissolves it. This compound is found in industrial ferro-silicons containing 10 to 20 per cent of silicon. To these it communicates its properties. Those compounds containing more than 15 per cent of silicon are only very slightly attacked by nitric acid, unless finely powdered.

A New Product of Tartaric Acid.—L. J. Simon.—During the calcination of tartaric acid in presence of potassium bisulphate, a new acid is produced, in addition to pyruvic and pyrotartaric, which the author succeeds in isolating. The crystals, when purified by crystallisation from hot alcohol, melt at 164° , after becoming greasy towards 158° . They re-solidify spontaneously at 156° . This new compound sublimes easily when heated to perfectly white needle shaped crystals or transparent plates. It is moderately soluble in boiling water and crystallises out on cooling. It is soluble in ether and acetic acid. The substance is a feeble acid, neutral to helianthine but acid to phthalein. The author also prepares its potassium salt, $\text{C}_7\text{H}_5\text{O}_3\text{K} \cdot 2\text{H}_2\text{O}$, the acid itself having a composition represented by $\text{C}_7\text{H}_5\text{O}_3$, and so being unsaturated. Bromine can be fixed in the cold. The potassium salt gives with silver nitrate a yellowish gelatinous precipitate, with lead acetate a white, and with copper acetate a bright green precipitate. This acid is isomeric with pyrotartaric acid, but is certainly distinct from it, and the author believes it to belong to the group of the furfurane acids.

Acetyl Derivatives of Cellulose and Oxycellulose.—Léo Vignon and F. Gerin.—Acetic anhydride, purified cotton dried at 110° , and pure dry zinc chloride, ZnCl_2 , when reacting together, give a product containing a white powder which is insoluble in water, infusible, insoluble in ether, very slightly soluble in alcohol, very soluble in acetic acid, acetone, acetic ether, and chloroform; the yield is about 25 per cent. The same operation can be repeated with oxycellulose, and there is formed, under the same conditions of acetylation, 40 per cent of the purified product, which decidedly reduces cupro-potassic solutions. A further acetylation may be performed by heating together crystallised acetic acid, purified cotton, and zinc chloride for twenty-four hours at boiling point. About 26 per cent of the product is thus obtained. These three products have

been analysed and found to be almost identical. As a result of various analyses they have been called triacetyl products, but estimation with acetic acid causes them to be classed between the tri- and the tetra-acetyl products and nearer to the latter.

MISCELLANEOUS.

University College, London.—We are requested to announce that a course of Lectures will be delivered by Dr. W. Hampson on Friday Evenings during November, December, January, and February, at 5.30 p.m. The first lecture will be delivered on November 9th. The subject of this course of lectures will be on "Methods of Producing Artificial Cold," and will be illustrated by experiments. Among the subjects involved will be early investigations on the condensation of volatile vapours; pressure in relation to boiling-points and freezing-points; critical temperatures; early commercial apparatus; later apparatus; intense refrigeration, with reference to the work of Piéet, Cailletet, Wroblewski, Dewar, Siemens, Solvay, Hampson, and others.—There will also be a course of eight Lectures dealing with the "Methods of Spectroscopy, especially in connection with the Photography of the Spectrum," by Mr. E. C. C. Baly, on Friday Evenings, at 5.30 p.m., commencing on January 18th, 1901.

NOTES AND QUERIES.

* * Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Assimilation of Nitrogen.—Will any reader be so kind as to give me the name of any recent publication or report bearing upon the question of assimilation of nitrogen by other plants than the Leguminosae, and by other organs than the nodules on the roots? I am convinced that these nodules are merely stores of nutriment. I have often dug up small vetches in gardens, and found, if they were extra luxuriant, that they had been growing above bits of dung put in for potatoes and other crops, and that the nodules were most abundant on the roots nearest the dung, and sometimes both above and below in close proximity to it, while they were but sparingly seen on the other roots; also, if a piece of dung was at the side of a plant, the roots on that side had nodules abundantly. The late Sir John Bennet Lawes found nodules on grasses as well as vetches.—JOHN MILNE, LL.D.

MEETINGS FOR THE WEEK.

MONDAY, 5th.—Royal Institution, 5. General Monthly Meeting. Society of Chemical Industry, 8. "Preliminary Examination of Applications for Patents," by W. Lloyd Wise. "The Early Manufacture of Sulphuric and Nitric Acids," by Oscar Guttman.

WEDNESDAY, 7th.—Society of Public Analysts, 8. "The Determination of the Available Brewing Extract of Malt," by Lawrence Briant. "The Definition of the Genuine Product," by C. E. Cassal, F.I.C. "Notes on certain B.P. Tests," by C. G. Moor, M.A., F.I.C., and Martin Priest.

Geological. "Additional Notes on the Drifts of the Baltic Coasts of Germany," by Prof. T. G. Bonney, D.Sc., LL.D., F.R.S., F.G.S., and the Rev. Edwin Hill, M.A., F.G.S. "On certain Altered Rocks from near Bastogne, and their Relations to others in the District," by Miss Catherine A. Raisin, D.Sc.

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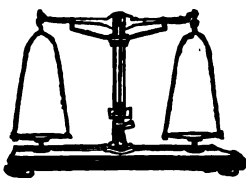
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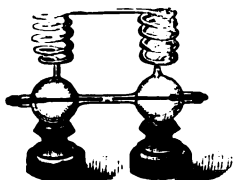
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THE MUTUAL RELATIONS OF IRON, PHOSPHORUS, AND CARBON, WHEN TOGETHER IN CAST-IRON AND STEEL.*

By J. E. STEAD, F.I.C., F.C.S.

UNTIL quite recently the accurate study of compounds containing iron and phosphorus has had little attention. Indeed such a study was practically impossible so long as there existed no micrographic methods for distinguishing the various compounds when associated together, and of separating them from each other by chemical or other means. At the time when the subject was first attacked by the writer, nothing whatever had been published dealing with micro-structure of metals containing free phosphide of iron.

Leopold Schneider (*Österr. Zeit. für Berg- und Hüttenwesen*, xv., p. 448) had previously published results proving that, in raw irons, free phosphide of iron could be isolated by treating them with cupric chloride, the phosphide being practically insoluble in that reagent.

No one has isolated the phosphide from wrought iron and steel, or proved that it existed as such in these metals, although it is generally admitted that what phosphorus was present existed in the definite chemical compound Fe_3P .

Before describing the nature of the researches made at Middlesbrough, it will be necessary to give a brief definition of some of the terms now in use in metallographic science.

Solid Solution.

The term solid solution has been defined by Sir W. Roberts-Austen as "a homogeneous mixture of two or more substances in the solid state, and solid solutions of metals when crystalline are solid isomorphous mixtures or mixed crystals."

This definition is a most concise one, and certainly appears to cover every kind of solid solution.

As a general rule, metals and their alloys, when solid, are masses of crystals, and, when one constituent crystallises with another to form a solid solution, such a crystal, in mineralogical nomenclature, would be termed isomorphous.

Many crystals homogeneously constituted, containing two or more crystalline substances, have not exactly the same crystalline form as either of the crystal components.

Professor Bauerman, in his work on "Descriptive Mineralogy," p. 338, says that, in the strict literal sense, the term "isomorphous" is only applicable to such substances as are cubic in crystallisation as in other systems: the relation between the members of isomorphous series is one of close analogy, but not of identity."

It would appear that the term solid solution is a more suitable general term, which may be applied to crystalline or non-crystalline substances, and conveys the meaning that the bodies in solid solution are as intimately associated as if in liquid solution, and that the liquid solutions become solid solutions when they solidify. In solid solutions no microscope can detect the substance dissolved, and in that respect are identical with liquid solutions.

The term "mixed crystal," which appears to have a continental origin, may be understood, by those who use the expression, to be synonymous with isomorphous crystal.

Mixtures are, as a rule, considered to be composed of separate substances mixed up together, but not constituting a homogeneous whole. Sir W. Roberts-Austen often refers to a eutectic being a mixture, and, as the constituents of eutectics are crystalline substances, a eutectic may be called a "mixture of crystals."

In the metals containing between 16 and 21 per cent phosphorus, and 84 and 79 per cent iron, there are crystals of Fe_3P and Fe_2P placed side by side, and anyone would be justified in saying they were composed of mixed crystals.

If the term mixture in metallography is used to mean, in one case, homogeneity, and in another heterogeneity, confusion is certain to follow.

The expression "fall out of solution" is often used by metallographers, and may mean that one of the constituents of an alloy separates in advance of the remainder when the metal is passing from the liquid to the solid state, or when a constituent separates after the metal has become solid. It does not necessarily mean that the constituent falls to the bottom of the cooling mass, and disentangles itself from the fluid matrix. In most cases the constituent which falls out remains suspended in separate isolated particles, and, when the mother-liquor solidifies, are retained in that position.

If the constituent is heavier than the mother-liquor, it falls to near the bottom; if it is lighter, it rises to near the surface.

Cementite is the mineralogical term given, by Professor Howe, for carbide of iron, Fe_3C .

Pearlite or *pearlyte* is another term for the pearly constituent of annealed steels first discovered by Dr. Sorby, of Sheffield. It consists of juxtaposed bands or grains of cementite, and iron alloyed with various metals and non-metals. It contains, according to circumstances, between 0.75 per cent and 1.0 per cent carbon.

The allotriomorphic crystals of Rosenbush are described hereafter as grains. They consist of crystal masses, which are so crowded together that the proper development of their crystalline faces and angles are interfered with.

ON THE MICROSCOPIC IDENTIFICATION OF THE PHOSPHORUS COMPOUND IN METALS.

The first object aimed at was to ascertain whether or not the phosphide isolated by chemical reagents by the Schneider process could be detected on the polished and etched sections of pig-metals containing it. With this in view, a specimen was selected which contained no chemically-combined carbon, and was what is known as glazed-Cleveland pig-iron, a variety sometimes accidentally produced when an excessive proportion of coke is charged into the blast furnace with the ore. It contained nearly 5 per cent silicon and 1.5 per cent phosphorus. On polishing the surface of a section, and etching it with dilute acid and afterwards examining it under the microscope, it was found to have distributed over it small patches or areas of a brilliant white constituent closely resembling massive carbide of iron.

It was easy to see that the bright parts remained brilliant, because the nitric acid used in etching had not corroded it.

In order to isolate it, another piece of the same metal was digested with dilute nitric acid in the cold, until apparently the chemical action ceased.

The residue was filtered off, and any phosphide of iron which might be in it was attracted from it by a magnet. This was ground to fine powder in an agate mortar, and was again digested with nitric acid to remove the inclusions which escaped the preliminary treatment. The final residue, after separating magnetically and washing with alcohol and ether, contained 15.1 per cent phosphorus.

Pure phosphide of iron, Fe_3P , contains 15.8 per cent phosphorus. It was therefore considered to be proved that the brilliant substance in the section was really phosphide of iron.

* Read before the British Association (Section B), Bradford Meeting, 1900.

Table of Heat Tints.

With steadily rising temperatures 200° to 400° C.				
Pearlite. 0·6 p. c. P; 0·75 p. c. C.	Iron.	Carbide of iron.	Phosphide of iron.	Sulphide of manganese.
White.	White.	White.	White, tinted yellow.	Pale lavender.
Very pale yellow.	Do.	Do.	Do.	Do.
Do.	Do.	Do.	Do.	Do.
Yellow.	Very pale yellow.	Do.	Do.	Do.
Do.	Do.	Do.	Do.	Do.
Yellow-brown.	Yellow.	Very pale yellow.	Do.	Do.
Do.	Do.	Do.	Do.	Do.
Brown.	Yellow-brown.	Do.	Do.	Do.
Do.	Do.	Do.	Do.	Do.
Red-purple.	Brown.	Yellow.	Very pale yellow.	Pale yellow.
Purple.	Red-purple.	Yellow-brown.	Do.	Do.
Blue.	Purple.	Do.	Pale yellow.	Do.
Do.	Blue.	Brown.	Yellow.	Do.
Pale blue.	Do.	Do.	Salmon.	Do.
Do.	Pale blue.	Red-brown.	Heliotrope.	Brownish white.
Do.	Do.	Red-purple.	Greenish.	Do.
Pale pea-green.	Do.	Purple.	Yellow.	Do.
Do.	Pale pea-green.	Blue.	Do.	Do.
Pale yellow.	Pale yellow.	—	—	—
White.	White.	—	—	—

A series of grey metals was then prepared containing varying proportions of phosphorus ranging from 0·03 per cent to 1·5 per cent. These were polished and etched, and examined under the microscope. As was to be expected, the relative proportion of the brilliant constituent was found to diminish incidentally with the phosphorus present. On removing the marks from the slides and disarranging their order, it was easy, after examination, to re-arrange them in consecutive order. Indeed, by a little experience, it was found that, by the micro-appearance, it was possible to approximately estimate the actual proportion of phosphorus present in grey metals.

Attention was next directed to the structure of white pig-irons containing phosphorus. In such material, practically all the carbon exists in the definite chemical compound Fe_3C (carbide of iron), which, as is well known, is practically unattacked by dilute acid, so that in the etched sections both the carbide and phosphide remain bright, and cannot be distinguished from each other.

It was found, however, that carbide and phosphide of iron take different tints on heating in presence of air, and that by careful heating it was possible to get the carbide coloured a deep red-brown when the phosphide was a pale yellow. The simplest way to obtain these tints is to place the brightly polished specimen upon the surface of fluid tin maintained at a temperature of 270° to 300° C., and remove it as soon as the tint of the specimen has assumed a purple tone to the naked eye.

The accompanying table gives at a glance the progressive colouring of iron, pearlite containing 0·6 per cent phosphorus and 0·75 per cent carbon, carbide, and phosphide of iron, when heated steadily from 200° to 400° C.

If the heating is done too rapidly, the graduations do not appear in the exact order as shown.

When phosphoretic white irons were examined after proper heating, it was easy to distinguish the carbide from the phosphide.

The method of heat tinting was first used by Professors Martens and Burhens, but they did not carry their investigations far enough to enable them to use it in differentiating carbides and phosphides. M. Osmond has, however, informed the writer that he has been able to detect phosphides in pig-metals by its use, but had not published his results. It is clear, then, that the value of the method has been independently proved in France and England.

CHEMICAL METHOD FOR DETERMINING FREE PHOSPHIDE OF IRON.

The method of Schneider for separating free phosphides is objectionable, as there is much trouble in getting the

precipitated copper dissolved, and also because some of the phosphide which is in solid solution yields a decomposition product which remains insoluble together with the free phosphide.

A similar product remains on dissolving the metals in either dilute hydrochloric acid or sulphuric acid. It was, however, found that nitric acid of sp. gr. 1·20, at a temperature not exceeding 20° C., does not, to any material extent, act upon phosphide of iron in the free state, but completely dissolves all of that compound which is in solid solution.

A chemical method of determining the free phosphide was thus discovered, which, although not as accurate as could be desired, nevertheless yielded results within 5 per cent of the actual proportion present.

Having thus at command microscopical and chemical methods by which free phosphide can be detected and determined, it became practical to make a systematic research, which previously was impossible.

COMPOUNDS OF IRON AND PHOSPHORUS.

These may be divided into four classes, as follows:—

CLASS I.,	containing between	traces	and	1·75	per cent.
II.,	"	"	"	1·75 p. c.	" 10·2 "
III.,	"	"	"	10·2 "	" 15·58 "
IV.,	"	"	"	15·58 "	" 21·68 "

CLASS I.—Phosphorus, Traces to 1·75 per cent.

The metals of this class consist of solid solutions of phosphide of iron in iron. The phosphorus is homogeneously distributed in each, and the microscope is incapable of detecting any independent separate constituent. On treating with hydrochloric or sulphuric acid part of the phosphorus is evolved as phosphine gas, PH_3 , and part remains in a soot-like amorphous residue. The proportion evolved in the gas depends partly on the strength of the acid, but mainly on the phosphorus present. When the iron is nearly saturated with the phosphide a very small relative quantity of phosphorus as PH_3 is formed, not exceeding 5 per cent of the whole; whereas iron with under 0·10 per cent phosphorus, *ceteris paribus*, yields from 50 to 70 per cent of that element in the gaseous condition.

The results of M. Osmond, who was the first to employ this method of research, generally coincide with these observations.

It is difficult to explain why a weak solid solution should yield to acid more PH_3 than a stronger one. We know, however, that more nascent hydrogen is available in the former case, and that, as the phosphide is more attenu-

ated, it is probably more easily decomposed and hydrogenised.

The brittleness of iron increases with the phosphorus, and as the relative proportion of the phosphine PH_3 gas formed on solution of the metals in acid decreases, as a rule, with the brittleness and the proportion of phosphorus, it would not appear that a high yield of the gas can be accepted as necessarily indicative of a condition of weakness, a conclusion contrary to that of Baron Juptner. It is the main facts, however, which it is necessary should be grasped at present; the more difficult problem may be left for future research.

The black insoluble residue has a complicated constitution. It ignites spontaneously on drying at near 80°C ., and burns with a phosphoretic flame, a property probably caused by the presence of a liquid or solid hydride of phosphorus.

The ratio of iron to phosphorus does not correspond to any simple atomic proportion. It is not attracted by a magnet; it contains water, and loses considerably in weight on calcining in an inert gas.

A similar compound is left on the long continued action of dilute hydrochloric acid on phosphide of iron when the dissolving is effected with free exposure to the air; one of the facts which led to the conclusion that the black residue in both cases had a similar source, that it was derived from phosphide dissolved in solid solution in one case, and from free phosphide in the other.

The solid solutions of iron and phosphide increase in hardness with each addition of phosphorus. When iron is saturated with 1.75 per cent phosphorus, it takes a well-hardened tool to drill it.

If the phosphorus is raised above 1.75 per cent, the excess separates when the metal solidifies as a eutectic composed of phosphide of iron, and iron saturated with phosphide of iron, and is found more or less perfectly enveloping the crystalline grains, or in irregular-shaped areas inside the grains. When iron containing 1.8 per cent phosphorus is annealed by long heating at about 900°C ., and is slowly cooled from that temperature, a considerable quantity of the phosphide falls out of solid solution and appears as prismatic crystals, and the matrix then contains only about 1 per cent of phosphorus in solid solution, an observation which disposes of the conception that the compound saturated with phosphide at its solidifying point is a definite chemical compound.

The following analysis shows the changes which were effected by annealing:—

	Carbon.	Phosphorus as free Fe_3P .	Phosphorus in solution.
	Per cent.	Per cent.	Per cent.
Original ingot	0.18	0.51	1.31
After passing through malleable castings furnace ..	Trace	0.88	1.06
Portion of last metal heated to 1100°C	Trace	0.95	1.01
Portion re-melted	Trace	0.20	1.74

It is interesting to note that the more or less perfectly formed idiomorphic crystals in the annealed metal must have been formed at a temperature below the melting-point of the eutectic. In other words, that the molecular movements resulting in such a marvellous re-arrangement of the phosphide of iron and matrix must have taken place when both were in the solid state. It affords another very important link showing how closely connected are solid and liquid solutions.

CLASS II.—Phosphorus, 1.75 to 10.2 per cent.

As before stated, when the phosphorus is increased above 1.75 per cent, the metal, in solidifying, throws off the excess which appears in the cold metal as a eutectic. The proportion of this steadily increases with the phosphorus, until, when 10.2 per cent is present, the whole mass

consists of the eutectic. It may be considered to have the constitutional analysis of—

Phosphide of iron	61 per cent.
Iron saturated with phosphide in solid solution	39 "
	100 "

The melting-point is about 980°C .

CLASS III.

When the phosphorus is increased above 10.2 per cent, the excess appears at first as well-formed crystals of Fe_3P , embedded in the eutectic, and when the quantity is greater it appears in the form of grains surrounded by the same substance. When 15.58 per cent is reached, the mass is homogeneous, and consists entirely of Fe_3P .

CLASS IV.—Phosphorus between 15.58 per cent and 21.68 per cent.

When the phosphorus is increased above 15.58 per cent, somewhat porous ingots result, and, until 21.68 per cent is present, it is easy to separate the powdered metal into two distinct parts.

On endeavouring to colour or stain the constituents of polished sections so as to identify them by the usual etching methods, no success followed the effort; but on heating them till a blue oxidation temper tint was just visible to the eye, on examination under the microscope two differently coloured parts became evident, one blue, more readily oxidised than the other, and a second which appeared yellow.

Both constituents were practically homogeneous, and were evidently definite chemical compounds. One of these compounds was less soluble in nitro-hydrochloric acid than the other. The more insoluble part was least readily attracted by the magnet.

The constituent readily coloured blue by heating was the more soluble in acid, and was strongly attracted by the magnet. It consisted of Fe_3P .

The more insoluble compound, Fe_2P , separated by acid, contained—

Iron	78.30 per cent.
Phosphorus	21.50 "
Not determined	0.20 "
	100.00

The compound of iron and phosphorus containing about 18 per cent phosphorus, which had about equal parts of Fe_3P and Fe_2P , was most fragile and very porous, and the crystals loosely attached to each other were easily separated by slight pressure. On placing the coarse crystalline matter on a steel plate, and heating it till some of the particles took a "temper blue colour," and then rapidly cooling the whole, it was easy to pick out the blue particles from those of a pale yellow colour. On testing them chemically side by side, the blue particles were found to approximate to the composition of Fe_3P , the yellow crystals to Fe_2P .

When the mixed mass of blue and yellow particles was approached by a magnet to within a distance of 2 m.m., the blue particles were attracted and adhered to the magnet, the yellow ones remaining behind.

The analyses of the magnetically separated compounds were as follows:—

	Magnetic.	Slightly magnetic.
Iron	84.00 per cent.	78.40 per cent.
Phosphorus	15.82 "	21.50 "
Not determined	0.18 "	0.10 "
	100.00	100.00
Corresponding to the formula of Fe_3P		Fe_2P .
(To be continued).		

THE ESTIMATION OF TUNGSTEN IN STEEL AND STEEL-MAKING ALLOYS.

By FRED. IBBOTSON and HARRY BREARLEY.

THE common way of separating tungsten from the solution of the steel in nitric, nitro-hydrochloric, or nitro-sulphuric acid is to evaporate to dryness, take up in HCl, and filter off the WO_3 at once, or after re-evaporating once or twice more. The avowed object of the re-evaporations is to ensure the complete separation of the tungstic oxide, some of which, with only one or two evaporations, is liable to remain dissolved in the HCl solution. These instructions are based on the very common assumption that the tungstic acid precipitated by the first evaporation remains as a precipitate while the second evaporation is being made. This, however, is not so; unless tungstic oxide has been ignited at redness, it is very appreciably soluble in hydrochloric acid, so that after evaporating any number of times the final "take up" in HCl always takes up also many times more WO_3 than was supposed to be unprecipitated at the first evaporation, and this is only re-precipitated after evaporating to low bulk or at once diluting with water. Tungstic oxide is hardly, if at all, soluble in HCl diluted with two or three times its volume of water.

Of the other processes adopted for separating the tungsten from the iron, the only ones much in use, which are not in some way modifications of those indicated above, are—

1. *Separation with Copper Solutions.*—In which case the residue may contain other non-combustible bodies besides tungsten, the most objectionable of which is chromium; and
2. *Solution in Acids and Fusion with Na_2CO_3 of the Dried Residue.*—In this case also chromium accompanies the tungsten. Even when the WO_3 is separated by evaporation with acids, it is occasionally contaminated with chromium.

Any of the above processes, when the WO_3 is freed from SiO_2 and Fe_2O_3 (traces) merely, or fused and re-precipitated as mercurous tungstate, give satisfactory results with pure carbon tungsten steels; the extreme results of estimations made on the same steel in seven different ways being 4.32 and 4.46 per cent tungsten.

The following process is submitted not as a rigidly accurate one; the results are always about 0.05 per cent too low. It is, however, more rapid than any other method we know, and—either with or without a correction—the results are good enough for technical purposes. The course of the reactions are most clearly seen when applied to a ferro-alloy containing very little carbon.

Digest the powdered alloy or steel borings with strong HCl (about 100 c.c. for 5 grms. of steel) short of boiling; the iron is easily attacked, but the W is not. On adding only a few drops of nitric acid the ferrous changes to ferric chloride, and then the W becomes visibly attacked until the clear orange-coloured ferric chloride blackens again, showing that some ferrous chloride has been formed. By a repetition of these changes, using only rather more nitric acid than is needed to keep the iron wholly in the ferric state, the sample all passes into solution in a few minutes. This may appear contrary to the experience of those who have frequently decomposed tungsten steels and alloys with aqua regia, and noticed that tungstic oxide—usually brown and not pure—nvariably separates; but the explanation is to be found in the use of enough HCl to completely dissolve all the WO_3 , and no more HNO_3 —which decreases the solubility of WO_3 in HCl—than is necessary to effect the oxidation. If, now, the acid solution of the metal be boiled until WO_3 just begins to separate, then diluted with at least twice its volume of water and boiled, all the WO_3 except 2 or 3 m.grms. is precipitated. The ignited precipitate contains usually the bulk of the silica, which is driven off with HF, and traces of Fe_2O_3 , which may be allowed for

after fusing with Na_2CO_3 , filtering, and estimating the Fe by re-ignition and weighing. The essential points are:—To have enough HCl present, in as strong a form as possible, to hold the WO_3 in solution; to keep the temperature below boiling-point, so as not to weaken the HCl; and, after everything has dissolved, to have as little HCl as is practicable over and above that needed to keep the WO_3 in solution. The process can obviously only be tested on actual steels. The following results (uncorrected) were obtained:—

TABLE I.—Percentage Tungsten.

New process.	Standard process.
4.36	4.38
2.91	2.95
3.49	3.54
2.99	3.02
8.68	8.70
6.93	7.00

The last two samples contained 2.25 per cent chromium. After fusing with Na_2CO_3 to separate the small amounts of iron, the filtrates were observed to have a faint yellow tint. This was allowed for by acidifying, titrating with $FeSO_4$ and $KMnO_4$, and calculating the amount of Cr_2O_3 to be deducted. We have never found as much as 1 m.grm. Cr_2O_3 along with the tungstic acid.

Ferro tungsten Alloys.—These alloys may contain from 15 to 80 per cent of tungsten. They are incompletely soluble in 1:20 HNO_3 and in nitro-hydrochloric acid as it is usually applied. The decomposition is both rapid and perfect when the powder, preferably agate ground, is treated with HCl and a minimum of HNO_3 in the manner already indicated. One grm. of a 50 per cent alloy can be completely dissolved by using 50 c.c. HCl; when the W is higher, rather more, when lower, rather less HCl is needed. As the amount of material which can be conveniently handled is, at most, but 2 grms., the small quantity of WO_3 which is not precipitated on diluting the dissolved sample with water cannot be overlooked. By evaporating the filtrate from a number of ferro-tungstens, we have found that about 1 per cent of the total tungsten present generally remains dissolved.*

When an empirical correction of this kind cannot be tolerated, and a more complete analysis is desirable, the dissolved sample is evaporated in the water-bath, boiled with HCl which has been diluted three or four times, and the $WO_3 + S.O_2$ filtered off. This alternative is longer, but more accurate and less troublesome because it demands little or no attention, and the WO_3 is in a more easily filtered state; and, further, the small amount of Mo which is usually present will have completely passed into solution.

Self-hard alloys, which are ferro-tungstens containing considerable amounts of chromium, silicon, and manganese, are readily attacked by HCl and a little HNO_3 . Column 4 in Table III. represents the analysis of an alloy which is sold, along with the following instructions, for the making of self-hard steels:—"One-third alloy melted with two-thirds Swedish Bessemer."

Nickel-tungsten Alloys.—This alloy should not be opened up by fusion with soda carbonate. If finely powdered, it is attacked by fusion, but it is necessary to examine the undissolved oxide for tungsten. This method has also the disadvantage of separating chromium, which occasionally occurs in small quantity in these alloys along with the tungsten.

It is very easy to dissolve the powdered alloy in a mixture of hydrofluoric and nitric acids. The efficiency of both this mixture and the HCl with a little HNO_3 depends on the solubility of WO_3 in HF or HCl, but as WO_3 is more soluble in the former, a much less volume of it is needed. To the dissolved sample, from which the nickel

* In some cases, after standing all night, hardly a trace of WO_3 can be found by evaporating the filtrate.

has a strong tendency to separate in well-formed green crystals, add H_2SO_4 , evaporate to strong SO_3 fumes, dilute, filter off WO_3 , and weigh. The nickel in the filtrate—separated from iron or not as occasion demands—is estimated cyanometrically. A rapid estimation of the Ni, if only small amounts of iron are present, may be accurately made by dissolving as before, pouring at once into an excess of ammonia, and titrating a portion of the filtrate for Ni.

The use of HF for dissolving the sample prevents Si being estimated; it also causes a portion of the molybdenum to be volatilised. The more complete examination is carried out by dissolving (as for ferro-tungsten), evaporating, separating the $\text{WO}_3 + \text{SiO}_2$, and estimating Mo as PbMoO_4 , and nickel with KCN and AgI.

The Analysis of Tungsten-molybdenum Steels.—Tungsten can be separated from iron by pouring the solution into an excess of caustic soda, just as Mo and Fe are separated (CHEMICAL NEWS, lxxxi., 269), but the former separation is not nearly so easily made as the latter. It can be made, however, with perfect accuracy when test solution, made by adding standardised Na_2WO_4 to a ferric solution is poured into a considerable excess of vigorously agitated soda hydrate. But the nitric acid solution of an actual steel cannot be so easily resolved, although it is desirable that it should be, in order that the molybdenum and tungsten might be precipitated together as lead salts and separated in the manner described in a former paper (CHEMICAL NEWS, vol. lxxxi., p. 13). No doubt the separation of the Mo and W from the iron will be made in this way ultimately, but at present we find the following process to be more reliable and expeditious:—

Dissolve 2 to 5 grms. of the sample in the manner previously described in connection with W steels, evaporate to pastiness or to dryness, but do not bake, boil with dilute HCl (three or four to one), filter off WO_3 , and estimate Mo in the filtrate by separating the iron with NaHO and precipitating the Mo as PbMoO_4 . The process was tested by making duplicate analyses of tungsten-molybdenum steels, by assaying mixtures of tungsten and molybdenum steels, and W steels mixed with fused metallic Mo. The results obtained were:—

TABLE II.

Percentage present.		Percentage found.	
Mo.	W.	Mo.	W.
1.16	4.50	1.18	4.51
2.51	4.50	2.54	4.47
5.67	4.50	5.70	4.51
1.41	4.38	1.40	4.38
Duplicate assays of		1.76	1.82
Mo-W steel ..		1.78	1.84

The Analysis of Tungsten Powders.—The points to which our attention has been directed in the examination of these powders are:—The estimation of W, Na_2WO_4 , WO_3 , Mo or Mo compounds, Si or SiO_2 , Fe and Fe oxides, Al_2O_3 , and CaO. The last two compounds occur more frequently and in larger amount than is commonly suspected.

Another constituent of these powders which has not, to our knowledge, been previously mentioned, and which, up to the present, we have no reliable means of estimating, induces us to defer what observations we have to make on the general analysis of these powders. This strange component exists as cubes and tetrahedra with colours varying from bright bronze to brown. In the purest form in which it has been isolated from the W powder, it has a specific gravity of 7.3. In physical properties therefore it corresponds to tungsten-bronze; and such of its chemical properties as we have determined favour the same conclusion. It need only be assumed that the alkali of the tungstate which is decomposed to provide the WO_3 for reduction to W is not washed completely away in order to provide, in the manufacture of the tungsten powder,

just such circumstances as are favourable to the production of tungsten-bronze.

The occurrence of these red crystals has been observed in each of twenty samples, taken promiscuously from British, German, and French supplies when examined in the following manner:—Boil with a dilute solution of NaHO in a basin, stir well, and pour the liquid off. By virtue of their lesser specific gravity, the coloured crystals lie on the surface near to the lip of the basin. A commercial sample made in this district contains nearly 10 per cent of these crystals. They are isolated by passing the powder on to a sieve of 60 meshes to the lineal inch; this retains 50 to 70 per cent of the impure crystals. These are boiled with NaHO, and washed so as to remove adherent powder. After drying, they are digested with nitrofluoric acid, which dissolves practically all the metallic tungsten, and also a portion of the crystals. The residue is washed and boiled with NaHO; the resulting highly coloured crystals have the aforesaid specific gravity.

We should be very pleased to hear from anyone who, having previously noticed these crystals, has identified them and knows how they may be accurately estimated.

The following are analyses of commercial tungsten alloys:—

TABLE III.

	Fe-W.	Ni-W.	Self hard.	W powder.
W ..	55.20	72.89	25.78	95.43
Mo ..	0.23	traces	—	0.16
Ni ..	—	22.96	—	—
Fe ..	42.59	1.26	55.99	0.22
Cr ..	—	—	2.90	—
Si ..	0.40	0.84	0.59	—
SiO_2 ..	—	—	—	0.40
C ..	1.09	1.32	4.64	0.05
Al_2O_3 ..	—	—	—	1.38
Mn ..	0.26	0.29	10.12	traces
S ..	0.165	0.275	—	—
Na_2WO_4 ..	—	—	—	1.25
WO_3 ..	—	—	—	0.67
Red crystals	—	—	—	Present, but not estimated.

99.935 99.835 100.02 99.56

The Technical Department,
University College, Sheffield.

THE REDUCTION OF TUNGSTIC ANHYDRIDE BY ZINC: THE PREPARATION OF PURE TUNGSTEN.

By MARCEL DELÉPINE.

FROM the calorimetric measurements made by M. Hallopeau and myself (Bull. Soc. Chim., Series 3, vol. xxi., p. 945) on the heat of oxidation of tungsten, it was easy to devise a method for the preparation of this metal; if its heat of oxidation is correct, all metals with a greater heat of oxidation would be able to remove it from its oxides in the same manner as it displaces from their oxides all the metals with a lower heat of oxidation.

Apart from the experiments made by M. Moissan (Comptes Rendus, 1896, vol. cxxiii., p. 13), which require a great deal of attention in order that the reduction of the tungstic anhydride by the carbon in the electric furnace may be stopped exactly at the phase when the metal is integrally decarburised, there exists up to the present time no process by which large quantities of pure tungsten can be prepared. The reduction of WO_3 by aluminium, according to M. Goldschmidt's process, even with the modifications introduced by M. Stavenhagen, gives the melted metal, but this latter is liable to contain aluminium (Berichte, vol. xxxii., p. 1513).

By the method I propose to use, I reduce tungstic anhydride by means of zinc, a common metal which adds a practical interest to the theoretical one, which is by no means to be disdained.

According to thermochemical data we have:—



As a matter of fact I have effected this reaction with: 1) pure zinc and pure tungstic anhydride; (2) pure zinc and pure tungstate of ammonium; (3) commercial zinc and tungstate of ammonium made from commercial tungstic anhydride; and (4) commercial zinc and tungstic anhydride such as is extracted directly from wolfram by Woehler's method.

It is sufficient to heat the mixture of WO_3 with 1.5 parts of powdered zinc to a red heat over a fire made of wood charcoal, coke, or gas, until the zinc no longer distils over; in all cases we obtain a black, very friable mass, consisting of tungsten, oxide of zinc, and a little tungstic anhydride, or a lower oxide, resulting from an incomplete reaction.

This mass should be treated in the following manner. The oxide of zinc formed is removed by hydrochloric acid, then wash till the acidity has been removed; this gives a product containing 98.5 per cent of W, or about 94 per cent of the metal, and 6 per cent of WO_3 . Boiling the black powder obtained, with soda for a few minutes, then washing until the alkalinity is removed, almost completely eliminates the oxides, and, according to the care with which the operation has been carried out, gives a metal of 99.5 to 100 per cent purity. In any case, the metal heated to redness for an hour in a current of hydrogen will always give a product of 99.8—99.9—100 per cent of purity. This operation can be carried out on quantities of two hundredweight at a time, which is impossible when starting from tungstic anhydride.

The following analytical results show the degree of purity of the products formed by the different methods of preparation mentioned above; 100 parts contain in tungsten:—

	After washing with HCl.	After using NaHO .	After the action of H.
1.	98.6	99.65, 99.44, 99.48	99.92, 99.89
2.	98.5	—	99.80, 99.93
3.	—	99.88, 100.00	—
4.	98.62	—	99.98, 99.90, 100.0

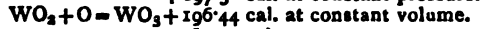
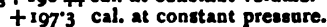
We see that the purity of the zinc has no influence on the quality of the product, which shows that these impurities were either volatile or soluble in the alkali. It must, however, be noticed that products 2 and 3 prepared from ammoniacal salts may possibly contain a thousandth part of nitrogen eliminable by means of the melted alkalis in the form of ammonia.

The metal obtained by this zinc process is a greyish-black dense powder, taking a metallic lustre on being subjected to great pressure or on trituration in a mortar, burning in the air with great facility, while becoming entirely changed into yellow tungstic anhydride. With other reagents it also shows considerable chemical activity, which, however, is only due to its fine state of division. Most of the above analyses were made by burning the metal placed in a heated crucible in the air; the oxide formed is completely soluble in melted carbonate of soda. By estimations with mercurous nitrate I recovered, to within 1/800th part, the WO_3 existing after the oxidation in the air. This slight loss may easily be attributed to losses of manipulation; the simple oxidation in the air constitutes, in fact, a very exact method of estimation.

The density of samples 2 and 4 was found to be respectively 18.67 and 18.61, figures which are very close to those given by M. Hallopeau for crystallised tungsten (*Bull. Soc. Chim.*, Series 3, vol. xix., p. 997), and by M. Moissan for melted tungsten (*loc. cit.*).

Finally, the finely-divided pure metal which I now possessed in large quantities enabled me to determine

afresh, and under more stringent conditions, the heat of oxidation of W to WO_3 . By operating on about 2 grms. I found for 1 gm. 1064.0 cal., 1071.5 cal., and 1067.2 cal., a mean of 1067.6 cal., or almost indidentically the same value that had been previously found, but certainly more exact. I, therefore, definitely put forward the following figures:—



These figures, so close to those for iron, suggest another question. Is it really necessary to heat to such high temperatures as those recommended by Deville, Riche, and Dumas to reduce tungstic anhydride by hydrogen? No. I have observed that tungstic anhydride heated in a porcelain boat in a glass tube loses all its oxygen in from one to two hours under the influence of dry hydrogen, and that at only a red heat considerably below the softening point of glass; the loss of weight is found to be equal to the increase of weight on oxidation.

To sum up, the reduction of tungstic anhydride, either by hydrogen or by zinc, easily enables us to obtain pure tungsten, and that in as large quantities as we wish, and by the use of zinc at temperatures very little above the distilling point of this metal. Apart from its pulverulent physical state, the metal thus prepared has the density and the heat of combustion of tungsten, either crystallised or melted; it can also, by compression or trituration, assume the brilliant lustre of a metal, so that we are perfectly justified in stating that it is in the same condition, its finely-divided state being only due to the want of fusibility of the metal.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 16, p. 675.

THE DETECTION OF IODIC ACID IN THE PRESENCE OF CHLORIC, BROMIC, PERCHLORIC, AND PERIODIC ACIDS, BY MEANS OF SULPHATE OF MORPHINE.

By C. REICHARD.

WHEN we treat an iodate, such as iodate of potassium, in aqueous solution, with a solution of sulphate of morphine and a few drops of sulphuric acid, we obtain a brown precipitate if we use a concentrated solution. If we have to deal with dilute solutions, a brownish colouration or a yellowish-brown or yellow precipitate is formed, according to the amount of iodic acid present. The reaction is more especially sensitive if, after adding the reagent, we drop a small quantity of ammonia into the mixture.

Sulphate of morphine enables us to detect the least trace of iodic acid, even in the presence of chloric, bromic, and perchloric acids, on which this reagent has no action. As for periodic acid and the periodates, the action is not the same, and we offer the following remarks on the subject. Periodate of potassium is very slightly soluble in water (100 parts of cold water dissolve 0.3448 part of KIO_4), and the aqueous solution treated with sulphuric acid remains colourless; but when treated with sulphate of morphine it almost immediately acquires a yellow colouration, and this phenomenon takes place as well with concentrated solutions as with dilute ones. After standing for a more or less prolonged period this colouration disappears, exactly as with iodate of potassium; but when we add a slight excess of dilute ammonia to either concentrated or dilute solutions of the periodate, there is, in both cases, a more or less pronounced colouration, but when left to themselves for a time at the ordinary temperature the two solutions behave differently. While

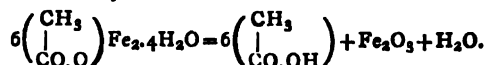
the dilute solution keeps its original brownish-yellow tint, even after several days, the concentrated solution becomes almost colourless after only twelve hours. The colour of this latter is brought back by ammonia, but it is then much less pronounced. The dilute ammoniacal solution, even when boiled, retains its colouration.

Solid iodate of potassium, treated with sulphuric acid and morphine, gives a solid brown precipitate of free iodine, while under the same conditions the periodate only gives a yellow solution. The very slight solubility of the periodate, compared with the great solubility of the iodate, enables us to separate these two iodic acids by means of successive additions of small quantities of cold water. In this manner we finally obtain a residue of pure periodate which can be easily recognised.—*Chemiker Zeitung*, 1900, p. 644.

A METHOD FOR THE ESTIMATION OF ACETIC ACID.

By V. DELFINO and M. MIRANDA.

WHEN we add an excess of a concentrated solution of ferric chloride to acetic acid (vinegar should be first decolourised by means of animal charcoal), a deep red colour is produced. This colouration is destroyed by heat; a gelatinous mass is then formed, which almost immediately resolves itself into hydrated ferric oxide, while acetic acid is set at liberty:—



(Marignac's formula).

When submitted to the action of heat the hydrated ferric oxide is transformed into anhydrous ferric oxide, which adheres to the sides of the beaker or flask in which the operation has been performed. It is then only necessary to determine the weight of the anhydrous oxide and to multiply the quantity of metallic iron obtained by the factor 3.2143 to get the quantity of acetic acid.

It is, however, simpler to estimate the metallic iron directly, and the following is the method we adopted:—The ferric oxide is treated with hot concentrated sulphuric acid, which transforms it into ferric sulphate,—



This solution is diluted, and a strip of silver is plunged into it. On applying heat, the silver causes the ferric sulphate to pass into the ferrous state, and at the same time it is itself dissolved, forming sulphate of silver,—



Care must, however, be taken not to allow the solution to cool, or else the sulphate of silver will be precipitated in the crystalline form and the ferric sulphate regenerated. After making sure that the reduction of the ferric salt is complete, we add an excess of hydrochloric acid to the solution to precipitate the silver in the state of chloride, which may then be separated by filtration. Under these conditions there is no longer any danger in allowing the filtered solution to cool, and the iron may then be estimated volumetrically by means of permanganate of potassium.—*Moniteur Scientifique*, Series 4, vol. xiv., October, 1900.

Royal Institution.—The Annual Course of Christmas Lectures, specially adapted to Young People, at the Royal Institution, will be delivered by Sir Robert S. Ball, F.R.S., Lowndean Professor of Astronomy in the University of Cambridge, whose subject is "Great Chapters in the Book of Nature." The first lecture will take place on Thursday, December 27, at Three o'clock, and the remaining lectures will be delivered on December 29, 1900, and on January 1, 3, 5, and 8, 1901.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 217).

IV. PURIFICATION OF THE MATERIAL.

It was not essential for the present investigation that the last traces of impurity should be removed; hence no attempt was made to prepare absolutely pure selenium. The substance in its original form contained a considerable quantity of sulphur and a very little tellurium as impurities. The method used was to dissolve by heating in concentrated sulphuric acid, thus oxidising the selenium to selenious acid, and then to precipitate by means of sulphur dioxide. Before precipitating, the sulphuric acid solution was diluted with water, and, if necessary filtered.

Divers and Shimose (1884) found that this method of purification was effective in removing the tellurium which does not come down under these conditions, but may afterwards be precipitated by the addition of hydrochloric acid.

After reduction, vigorous shaking is necessary to bring the material together, and it should then be washed until the washings show no further acidity. If the red selenium be then separated on the filter, it will be found very difficult to dry; standing for weeks in a desiccator over sulphuric acid will not free it from water; it is therefore better to make a final washing with alcohol, then filter and dry in the air on a porous porcelain plate. One gram of the material thus purified, when dissolved in nitric acid and diluted, gave no appreciable indications of the presence of tellurium, and a test for sulphur showed that this element also was present in only very small quantity.

V. THE PROPERTIES AND TRANSFORMATIONS OF THE DIFFERENT FORMS OF SELENIUM.

1. The Vitreous Modification.

The form in which the element comes into commerce is the one known as vitreous, from its brilliant conchoidal fracture. The commercial material in former years consisted of medallions bearing the image of Berzelius, the discoverer of the element, but it is now almost always supplied in rods. Its colour in the mass is black, but thin fragments are translucent, and display a deep ruby-red colour, and when very finely powdered the powder is red. When heated it begins to soften at about 50°, and if the heating is extremely rapid it is possible to raise the temperature to 220° without causing any transformation into a denser form; at this temperature it is distinctly liquid, though still viscous; it does not attain a state of thin fluidity below about 250°. This has led to a misleading statement in many of the text-books, the responsibility for which rests with Sacc (1847) to the effect that, while the grey crystalline or metallic variety melts sharply at 217°, the vitreous form only becomes really liquid at 240°—250°. The fact, of course, is that the liquid obtained at 217° is the same from whichever source, but it possesses at that temperature a moderately high viscosity. When melted selenium is cooled down the vitreous form is always obtained, unless the cooling is conducted very slowly, in which case it may go over to the more stable metallic form.

Berzelius (1818) remarks that the vitreous form is semi-fluid at 100°, completely so a few degrees higher; probably in his experiment the transformation into the metallic form set in at about the time that the temperature had attained 100°; this change sets free so large a quantity of heat that the mass may very well have assumed for a few moments a state of thin fluidity.

If vitreous selenium be slowly heated and a thermometer placed in the mass, it is found (Regnault, 1856) that when the temperature has attained a point about 96°—97° there

* From the *Journal of Physical Chemistry*, vol. iv., No. 6.

is a sudden rise in the temperature, amounting under favourable circumstances to as much as 150° ; the mass becomes completely fluid, and after a few moments hardens again. The substance thus formed is found, when examined, to possess quite different properties from the original vitreous selenium. This is, in fact, the grey crystalline form, appropriately called by Regnault metallic selenium.

This transformation was studied by Hittorf (1851), later by Regnault and many others. Regnault's observation that the change begins at 96° – 97° has led to the belief that there is something significant in these temperatures. Such is not the case. The fact is that vitreous selenium probably goes over into the metallic form at all temperatures under 217° , but the change, in the absence of solvents does not possess a measurable velocity below about 90° . Indeed, Regnault states that it may be kept for hours at 90° without any change occurring. This is probably true, in general, when selenium is heated by itself; nevertheless, Hittorf states that the change occurs anywhere between 80° and 217° , and Rammelsberg (1876) gives 90° as the limit of stability. At 95° , or any higher temperature, until we approach the melting-point, the change goes on rapidly. Hittorf made some experiments to determine the point of maximum velocity, and placed it at about 125° .

Positive evidence was still wanting to show whether the change was really a reversible or an irreversible one, i.e., to determine whether vitreous selenium is really unstable at all points below 217° , or whether it has at lower temperatures a region of stability. The idea of a transition point has gained a hold on the minds of some, through a remarkable experiment of Lehmann, which we shall now consider. Thus Tammann (1897) has been led to consider this an example of a substance having a second melting-point, analogous to the case of chloroform as reported by Piçet (*Zeit. Phys. Chem.*, 1895, xvi., 422). Further, Le Chatelier, in a more recent article (*Comptes Rendus*, 1899, cxix., 282), expresses himself in these words: "Selenium is only stable in the crystalline state above 60° and below 214° . Outside of these extreme limits of temperature, the amorphous modification (vitreous, liquid) alone is stable."

The observation of Lehmann upon which these statements are based, was made with the microscope, and is reported by him as follows (*Molekularphysik*, i., 712):—"When melted under a cover-glass, selenium appears as a clear red transparent liquid; on further warming, the liquid gets darker and black points make their appearance here and there; these increase in size and assume the aspect of spherical aggregates (sphärolithische Aggregationen) of the insoluble grey modification. . . . The crystals soon fill the whole field, and, on still further heating, the mass melts to a dark almost opaque liquid. On cooling down slowly, the spherical crystals of the grey modification appear at first, and then, as they grow steadily smaller, the colour of the liquid becomes less intense, and we obtain, finally, the original clear red liquid. If the cooling is rapid, the crystals of the grey modification do not appear, but the liquid passes gradually from dark red to light red. The same behaviour is shown on rapid warming; here also the black crystals may appear momentarily and disappear again, but sometimes the transition is direct from the red condition into the more deeply coloured one. The increased absorption of light on heating and the reverse change on cooling, point to a chemical change in the liquid such that it is to be regarded at higher temperatures as partially a solution of the grey modification.

On attempting later to reproduce the above inversion (Rückgängigwerden der Entglasung) it did not prove possible to do so; perhaps because of greater or less impurity of the selenium, perhaps because the conditions were not favourable for the production of the grey (light-sensitive, less conducting) modification." All my attempts to repeat this observation of Lehmann's also failed. I have not been able to find under the microscope anything abnormal

in the behaviour of the element. Lehmann's observation must therefore remain for the present unexplained; possibly local heating or some other experimental irregularity may have been the cause of the appearance and subsequent disappearance, at lower temperatures, of the crystals.

A better method of approaching this problem is afforded by measurements with the dilatometer, and the following section is devoted to an account of such experiments.

Dilatometer Measurements.

The experiment of Lehmann, to which reference is made above, indicated the possibility of a real transition-point in the change from vitreous to metallic selenium. Dilatometric measurements being the most satisfactory means of establishing such a point, the present study was begun with the dilatometer. The difficulties met with, which are discussed later on, had their source in the fact that it was necessary to work over a range of more than 200° , and it was not easy to find a liquid which answered the purpose. Several were tried—*aniline*, *quinoline*, *glycerin*, and solid and liquid *paraffin*.

For those determinations which were made with solid paraffin, it was necessary to surround the stem of the dilatometer with a steam jacket, and also to enclose that part of it which passed through the lower stopper of the jacket, in copper tubing, to prevent the solidification of the paraffin in the capillary.

Even when liquid paraffin is used, the steam jacket is of value because, without it, the high viscosity of the paraffin at lower temperatures renders it difficult to obtain concordant results where the bulb is subjected to rapid changes through a wide range of temperature.

For the sunlight experiments, a bath of glycerin or high-boiling paraffin was used; for the others, a bath of oil. The Reichert gas regulator proved of service in maintaining a constant temperature within a couple of degrees, when it was desired to keep the bath for some time at a fixed point. The stirring was done by means of an electric motor; what is known as the Ajax motor proved most efficient. The dilatometer tube was always sealed; hence the pressure was generally higher than one atmosphere for the upper ranges of temperature. The recorded experiments are given under their original numbers, so that the series as given here is not complete.

(To be continued).

THE TITRATION OF MERCURY BY SODIUM THIOSULPHATE.*

By JOHN T. NORTON, Jun.

ACCORDING to J. J. Scherer (*"Lehrbuch der Chemie,"* i., 513) mercurous nitrate, mercuric nitrate, and mercuric chloride may be estimated by direct titration with sodium thiosulphate, $\text{Hg}_2\text{S}_2\text{HgS.Hg}(\text{NO}_3)_2$, and 2HgS.HgCl_2 , being the precipitates obtained in each case. I have been unable to obtain access to Scherer's original publication, but Sutton (*"Volumetric Analysis,"* p. 220) gives the following very general directions for this process:—

(a) *"Mercurous Salts."* The solution containing the metal as a protosalt only is diluted, gently heated, and the thiosulphate delivered in from the burette at intervals, meanwhile well shaking until the last drop produces no brown colour. The sulphide settles freely, and allows the end of the reaction to be easily seen. One c.m.³ of the 1/20 normal solution of thiosulphate = 0.02 gm. Hg or 0.0208 HgO.

(b) *"Mercuric Nitrates."*—The solution is considerably diluted, put into a stoppered flask, nitric acid added, and

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, 1900, vol. x., No. 35.

TABLE I.

	HgCl ₂ taken, calculated as Hg.	Na ₂ S ₂ O ₃ in excess.	Volume at beginning.	HgCl ₂ found, calculated as Hg.	Error.
	Grm.	C.m.s.	C.m.s.	Grm.	Grm.
1.	0.0446	46.28	200	0.0343	0.0103 -
2.	0.0354	46.28	400	0.0326	0.0028 -
3.	0.0356	44.97	400	0.0225	0.0131 -
4.	0.0345	44.5	100	0.0308	0.0037 -
5.	0.0154	44.43	50	0.0326	0.0028 -
6.	0.0382	22.59	50	0.0354	0.0028 -
7.	0.0375	8.58	50	0.0385	0.0010 +
8.	0.0371	1.84	50	0.0304	0.0067 -
9.	0.0731	2.28	50	0.0774	0.0043 +
10.	0.1486	9.34	50	0.1489	0.0003 +

TABLE II.

	HgCl ₂ taken as Hg.	Volume at be- ginning.	Temp.	Stand- ing.	Na ₂ S ₂ O ₃ in excess.	HgCl ₂ as Hg. Found.	Error.
	Grm.	C.m.s.	°C.	Minutes.	C.m.s.	Grm.	Grm.
1.	0.0738	50	36	40	16.68	0.0494	0.0244 -
2.	0.0741	50	70	15	15.42	0.0738	0.0003 -
3.	0.0741	75	70	12	16.07	0.0733	0.0008 -
4.	0.0744	50	70	10	14.6	0.0755	0.0011 +
5.	0.0764	50	72	7	6.77	0.0771	0.0007 +
6.	0.0762	50	75	10	8.54	0.0799	0.0037 +
7.	0.0756	50	73	15	9.99	0.0815	0.0059 +
8.	0.0774	50	68	15	10.84	0.0767	0.0007 -
9.	0.0745	75	69	7	6.62	0.0805	0.0060 +
10.	0.0736	50	68	5	15.82	0.0714	0.0022 -

TABLE III.

	HgCl ₂ taken as Hg.	Volume at be- ginning.	Temp.	Na ₂ S ₂ O ₃ in excess.	HgCl ₂ found as Hg.	Error.
	Grm.	C.m.s.	°C.	C.m.s.	Grm.	Grm.
1.	0.0749	50	70	4.15	0.0751	0.0002 +
2.	0.0749	50	75	0.72	0.0728	0.0021 -
3.	0.0756	50	72	1.46	0.0759	0.0003 +
4.	0.0753	50	70	2.57	0.0750	0.0003 -
5.	0.0390	50	70	3.43	0.0395	0.0005 +
6.	0.0388	50	72	8.19	0.0390	0.0002 +
7.	0.0380	50	76	2.03	0.0393	0.0013 +
8.	0.1494	50	78	4.99	0.1498	0.0004 +
9.	0.1489	150	78	4.38	0.1512	0.0023 +
10.	0.1480	50	70	0.52	0.1438	0.0042 -
11.	0.1498	50	78	1.47	0.1540	0.0042 +
12.	0.1484	50	71	2.09	0.1517	0.0033 +
13.	0.1480	75	72	1.59	0.1509	0.0029 +

TABLE IV.

	HgCl ₂ taken as Hg.	Volume at be- ginning.	Temp.	Na ₂ S ₂ O ₃ in excess.	HgCl ₂ found as Hg.	Error.
	Grm.	C.m.s.	°C.	C.m.s.	Grm.	Grm.
1.	0.0759	100	60	3.06	0.0766	0.0007 +
2.	0.0384	100	60	2.81	0.0387	0.0003 +
3.	0.1498	100	60	1.1	0.1500	0.0002 +
4.	0.1503	100	60	1.63	0.1506	0.0003 +
5.	0.1479	100	60	2.41	0.1480	0.0001 +
6.	0.1489	100	60	2.12	0.1503	0.0014 +
7.	0.2244	100	60	2.63	0.2259	0.0015 +
8.	0.1490	100	60	2.33	0.1484	0.0006 -
9.	0.0758	100	60	2	0.0762	0.0004 +
10.	0.0383	100	60	2.58	0.0379	0.0004 -

the thiosulphate cautiously added from the burette, vigorously shaken meanwhile, until the last drop produces no further precipitate. Scherer recommends that when the greater part of the metal is precipitated the mixture should be diluted to a definite volume, the precipitate allowed to settle, and a measured quantity of the clear liquid taken for titration; the analysis may then be checked by a second titration of the clear liquid if needful. One c.m.³ of 1/20 normal thiosulphate = 0.05 of Hg or 0.0162 of HgO.

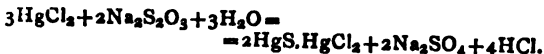
(c) *Mercuric Chloride.*—With mercuric chloride the end of the process is not so easily seen. The very dilute solution is acidified with hydrochloric acid, heated nearly to boiling, and the thiosulphate cautiously added so long as a white precipitate is seen to form; any great excess of the precipitant produces a dirty-looking colour. Filtration is necessary to distinguish the exact ending of the reaction. One c.m.³ of 1/20 normal thiosulphate = 0.015 Hg or 0.0162 HgO.

Fresenius ("Quantitative Analysis") gives practically the same directions, but omits all mention of that portion of the process dealing with mercurous nitrate.

In view, therefore, of the scant information available on the subject, and of the apparent difficulty of working the process accurately according to the directions given, an attempt was made to ascertain whether the careful regulation of temperature, dilution, and amount of acid present, might not produce beneficial results.

That portion of the process dealing with mercuric chloride was first taken up. The mercuric chloride used was pulverised, dried at 100°, and its purity proved by several determinations as mercuric sulphide. The sodium thiosulphate was made up of approximately 1/20 normal strength and standardised on decinormal iodine, which in turn was titrated against decinormal arsenious acid made from pure re-sublimed arsenious oxide.

For the action of sodium thiosulphate upon the mercuric chloride Scherer gives the equation—



According to my experience, the action results in the formation of a dense white precipitate which refuses to settle either by shaking or standing, thus making it impossible to fix the end reaction by reading the first drop of thiosulphate which produces no further white precipitate in the solution containing the mercuric chloride. Recourse must be had, therefore, to filtering. By far the quickest and neatest method is to use the asbestos filter deposited on a large perforated platinum cone (*Amer. Chem. Journ.*, i., 321). This cone is set in a glass funnel by means of a rubber connector, and the funnel is passed through the stopper of a large side-necked Erlenmeyer connected with an exhaust-pump. A little asbestos fibre shaken in the liquid to be filtered was found to be very beneficial in preventing the precipitate from running through the filter. In all the following experiments the thiosulphate was run into the solution containing the mercuric chloride in excess, the whole shaken up with asbestos fibre, filtered, and the excess of thiosulphate determined by 1/20th normal iodine. This method of procedure seems to be far preferable to attempting to catch the end of the reaction by running in the thiosulphate until the last drop produces no precipitate. In the experiments shown in Table I. no attention was paid to the temperature of the solution, and the thiosulphate was run in until the liquid turned brown. In every case the solution was allowed to stand until there was no further visible change of colour.

A glance at the table shows that the results are most irregular. In Table II. is seen the result of regulating the temperature, and the length of standing after the addition of the sodium thiosulphate.

These results, although better than those of Table I., are still very uncertain. On the supposition that the change from white to black, which takes place in the solution after the addition of an excess of sodium thiosulphate more or less quickly, according to the temperature, was due to an increased amount of HgS in the compound 2HgS.HgCl₂, the next step was to ascertain whether this could be avoided by stopping the addition of the thiosulphate at the first indication of a change of colour in the white precipitate, diluting the solution with a large amount of cold water, and immediately throwing it on the filter. The accompanying Table (III.) shows the result of the experiments.

In the case of quantities of mercuric chloride up to 0.1 grm. the results shown in Table III are very satisfactory, but when larger amounts of mercuric chloride are used the errors again become prominent. In Table IV. the effect of lowering the temperature to 60° C. and of increasing the dilution to 100 c.m.³ is shown.

From this table it is plain that Scherer's process for the estimation of mercury in the form of mercuric chloride is capable of yielding accurate results if carried out under certain fixed conditions. These conditions, which must be closely adhered to, are as follows;—The solution containing the mercury in the form of mercuric chloride is placed in a litre flask, diluted to 100 c.m.³, and heated to a temperature of 60° C. The sodium thiosulphate in 1/20th normal solution is run in from a burette until the white precipitate formed begins to take on a brownish tinge. The solution is then diluted with cold water, some asbestos fibre added to coagulate the precipitate, and the whole is quickly thrown on the filter. After careful washing of the precipitate, the filtrate is diluted to a definite volume, 3 grms. of potassium iodide added, and the excess thiosulphate titrated with iodine and starch solution. The duration of the process need not exceed fifteen minutes. It is worthy of note that there is no necessity of using any hydrochloric acid in addition to that formed in the reaction. This certainly eliminates one probable source of error—the interaction of hydrochloric acid and sodium thiosulphate.

(To be continued).

PROCEEDINGS OF SOCIETIES.

ROYAL INSTITUTION.

General Monthly Meeting, November 5th, 1900.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

Mr. W. H. Maw, Mrs. R. Middleton, and Dr. W. H. Perkin, F.R.S., were elected Members.

The special thanks of the Members were returned to Dr. Frank McClean, F.R.S., for his donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures, and to Dr. Rudolph Messel for his present of a Bronze Bust of Sir Humphry Davy.

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

October 30th, 1900.

Prof. HORACE LAMB, F.R.S., President, in the Chair.

A PAPER on "*The Solubility of certain Lead-Glasses or Fritts used in the Preparation of Pottery Glasses*," by WILLIAM JACKSON and EDMOND M. RICH, was read.

The paper described experiments carried out to determine what factors, apart from chemical composition, affect the amount of lead oxide yielded to dilute hydrochloric acid by lead fritts as used by potters. It was found that the solubility is increased in a very marked manner by increase of fineness, so that it appears that if details are given of the solubility of fritts they should be accompanied by particulars of its degree of fineness. The solubility of the same frit reduced to different degrees of fineness varied from 1 to 15 per cent of the material used. It was found also that after the action of the acid has proceeded for a short time it appears that the whole of the soluble lead has been extracted. This, however, was due, not to the absolute insolubility of the remainder, but to the formation of an insoluble layer on the surfaces exposed to the action of acid which protects the particles from further

action. By removing this layer by chemical or physical means it was found possible to extract more lead oxide from the frit, and, by continually removing this insoluble layer, it was possible to extract continually more lead oxide, until practically the whole of the lead oxide passed into solution.

A discussion followed the reading of the paper, in which the President, Mr. Wm. Burton, F.C.S., and Mr. T. Turner (Organising Secretary to the Technical Instruction Committee of the Staffordshire County Council) participated.

NOTICES OF BOOKS.

A Treatise on Qualitative Chemical Analysis. ("Traité de Chimie Analytique Qualitative"). By LOUIS DUPARC, EMILE DEGRANGE, and ALFRED MONNIER. Geneva: Henry Kundig. Paris: Félix Alkan. 1900. Pp. 223.

FOR the proper recognition of the chemical characteristics of any particular element, it is necessary to be thoroughly acquainted with its properties. With this object in view the authors have given *in extenso* all the reactions of the bases and acids, and have united the bases in four groups, established not so much on chemical analogies as on the wide methods of separation utilised in analysis. It would be useless to attempt chemical analysis without previously having acquired a general knowledge of inorganic chemistry, and especially of the atomic theory, the nomenclature of compounds, and of the mechanism of reactions and chemical equations. Without this preliminary knowledge practical work is apt to degenerate into purely mechanical operations, the student getting into the habit of adding so many drops from bottle No. 1, and so many from bottle No. 2, merely because the book tells him to, while he does not very often really understand what he is doing.

After devoting more than two-thirds of the book to what may be called preliminary work, such as a general examination of the materials and reactions utilised in analysis both by the wet and dry ways, detailed reactions of the bases and acids, the authors give a number of systematic tables for mineral analysis. These tables differ slightly from those ordinarily in use, inasmuch as groups 1 and 2 are combined so that there are only four groups in all. We can hardly see the necessity for this change, as the Hg, Ag, and Pb are, after all, separated from the rest of the usual second group, and then separated from each other in the usual manner.

The book closes with a graphic table of solubilities, a comprehensive table of contents, but no index.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxi., No. 16, October 15, 1900.

Preparation and Properties of the Carbides of Neodymium and Praseodymium.—Henri Moissan.—The oxides of neodymium and praseodymium, when heated in presence of carbon in the electric furnace, are transformed into the crystalline carbides of formulae NeC_2 and PrC_2 . These carbides decompose water in the cold, with production of a mixture of hydrogen carbides and the hydrated oxide. The author has already shown that the three carbides of the alkaline earths, prepared in the electric furnace, give, by their decomposition of water, only pure acetylene; on the other hand, aluminium carbide gives,

under the same conditions, only methane. It is known that neodymium and praseodymium belong to the cerium group, a group of metals placed, from consideration of its properties, between the metals of the alkaline earths and aluminium. It is curious to notice that neodymium and praseodymium carbide give, when placed in contact with water, a complex mixture of hydrocarbons, rich, however, in acetylene and methane. Further, it should be mentioned that the quantity of acetylene given by these different carbides diminishes from cerium to neodymium, and that neodymium and praseodymium, metals so closely allied as to have been for a long time confounded under the name of didymium, give with water a mixture of carbides of a very similar composition. Finally, the carbides of cerium, lanthanum, neodymium, and praseodymium correspond to the general formula RC_2 .

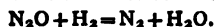
The Accessory Reactions of Electrolysis.—A. Brochet.—When a concentrated solution of sodium hypochlorite undergoes electrolysis and an estimation of the quantity of the salt left in solution is made, it is noticed that this quantity does not correspond to the theoretical number, more hypochlorite being decomposed than should be according to theory. The reverse is true in the case of the chlorate. At first sight this appears to be a case of non-accordance with Faraday's law, but experiment shows it to be the result of an independent reaction of the electric current. The author designates such a reaction as an accessory reaction of electrolysis. He investigates such reaction and finds that during the electrolysis of a solution of chloride the limiting value of the quantity of hypochlorite, due to oxidation and reduction, is 12.7 grms. of active chlorine per litre. He shows that if the reduction is suppressed this limiting value becomes 23.5 grms. The question then arises as to whether this limit is due only to the oxidation of the hypochlorite and if the accessory reaction is not the true cause. This question cannot be satisfactorily answered, owing to the difficulty of following the accessory reaction in the case of dilute solutions of hypochlorite. In all cases it is certain that, if the limit is due solely to the oxidation of the hypochlorite, it is useless to seek to suppress this oxidation, because a further check would be found due to limit of the accessory reaction.

Isopyrottritic Acid, a New Pyro-product of Tartaric Acid.—L. J. Simon.—In a former paper the author mentions an accessory product formed during the calcination of tartaric acid, which is an acid isomeric with pyrottritic acid, $C_7H_8O_3$, but distinct from it. He gives it the provisional name of isopyrottritic acid. The present paper contains an account of the properties and various reactions of this acid. When dissolved in water or an organic solvent, it gives with ferric salts, especially with the chloride, a very intense violet colouration. This colouration is very stable, not being alterable by time or heat. It is decomposed by strong acids, but on dilution will reappear if the acid is not present in too large quantities. Although it seems to be premature to attribute to this acid a definite composition from the facts mentioned in this and the preceding paper, it presents a certain analogy to salicylic acid, and so may be considered as a dihydrox-oxybenzoic acid, $C_6H_4H_2(OH)CO_2H$.

MISCELLANEOUS.

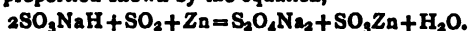
The Estimation of Nitric Acid in Gas Analysis.—G. von Knorre and K. Arndt.—It has been observed that when a mixture of nitric oxide and hydrogen in excess is passed through a capillary glass tube containing platinised asbestos, both nitrogen and ammonia are simultaneously collected, though in variable proportions, besides a certain amount of water vapour. The authors carried out their experiments at a much higher temperature in a platinum capillary tube heated to bright redness. Under these conditions, if the current is very slow, no ammonia is formed, and the reaction takes place according

to the following equation: $2NO + 2H_2 = N_2 + 2H_2O$, with the contraction of 1.5 times the volume of NO. In the same manner nitrous oxide reacts under similar conditions with an equal contraction of its volume,



By this means we are enabled to make analyses of the following mixtures:—NO with N_2O , NO with N, N_2O with N, N_2O with O, &c.—*Berichte*, xxxii., p. 2136.

Hydrosulphurous Acid.—A. Bernthsen and M. Bazlen.—One of the authors has previously (*Berichte*, vol. xiii., p. 2277) attributed the formula SO_2H or $S_2O_4H_2$ to Schutzenberger's hydrosulphurous acid instead of SO_2H_2 . The authors have now succeeded in preparing the neutral hydrosulphite of sodium in a state of purity. They treat powdered zinc with monosodic sulphite in the ordinary manner, and immediately add sulphurous acid in the proportion shown by the equation,—



Milk of lime is then added in slight excess, and the solution filtered. This filtrate contains only hydrosulphite of sodium. In this manner commercial solutions of this salt can be prepared at 15–16° B., of which 5 kilos. will suffice to reduce 5 kilos. of indigo. If such solutions are treated with sea-salt the hydrosulphite is precipitated in the pure state, in the form of crystals; the same will occur even if we add NaCl to the warm liquid and allow to cool. NaHO may also be used instead of NaCl. The analysis of the salt leads to the formula $S_2O_4Na_2 + 2H_2O$. It occurs in thin prisms with a vitreous lustre, 15 m.m. in length, easily oxidisable in moist air, but lasting for days without change in dry air, and fairly stable in dry air in a closed vessel. The dried salt burns at a dull red heat with a blue flame, and gives off SO_2 . This preparation was the subject of a German patent of the 23rd May, 1899, by the Badische Anilin und Soda-Fabrik. M. A. Nabl has recently (*Mon. f. Chim.*, 1899, p. 679) prepared hydrosulphite of zinc, S_2O_4Zn , in the solid state by the action of SO_2 on granulated zinc in absolute alcohol. M. Grossmann admits the existence of acid salts; they, like the free acid, should not be very stable, for the addition of a strong acid to the solution of the pure sodium salt gives a red colouration, quickly followed by a deposit of sulphur.—*Berichte*, vol. xxxiii., p. 126.

Distillation in Vacuo, and on some Regular Laws Obeyed by Liquids and Vapours in Vacuo.—F. Krafft.—When we boil, in vacuo, a substance having a high molecular weight, the operation proceeds in a very similar manner to distillation at the ordinary pressure, in this sense, that we observe a more or less thick layer of vapour above the surface of the liquid, according to whether we heat strongly or not, and quite distinct from the vacuum above, so that 2 or 3 c.c. above the limit cathodic rays may be made to pass. When this layer of vapour is very thin the temperature of the vapour is, to a fraction of a degree, the same as that of the boiling liquid, but if the layer is thick it exerts pressure on the liquid and retards its boiling, so that the upper layers of the vapour are several degrees cooler than the lower layers or than the liquid itself; other conditions being equal, however, the differences are proportional to the thickness of the column of vapour. The author has verified this with the fatty acids and with the saturated carbides. In the same series of homologues, the differences of temperature between the boiling liquid and the lower layers of vapour, at equal thicknesses of the latter, is proportional to the molecular weight. The author next deals with the difference between the fusing-points of substances (the fatty acids, the saturated carbides, the olefines), and their boiling-points in vacuo. For the same series of homologues it increases from one term to another in arithmetic progression, or, in other words, proportionally to the molecular weight. For the normal paraffins the rate of progression is 4.22°, for the saturated fatty acids 4.9°.—*Berichte*, vol. xxxii., p. 1623.

Conditions of Crystallisation of Colloidal Saline Solutions.—F. Krafft.—Alkaline soaps dissolved in warm water are deposited, on cooling, in a gelatinous form, and this deposit takes place at a temperature slightly lower than that of the fusing-point of the free acid. For the homologues of a high order, like palmitic or stearic acids, the difference is only a fraction of a degree. The author finds that the salt is entirely hydrolysed in aqueous solution, and that the fatty acid, although insoluble, is held in suspension in the state of a colloidal emulsion. This solution can only exist above the fusing-point of the acid. In the case of the lower homologues, the solubility of these latter lowers the point of solidification. Another cause of this lowering exists in the affinity of the alkali for water; the potash soaps remain gelatinous at lower temperatures than the soda soaps. The author has also examined the solidification of aqueous solutions of salts formed of a strong acid united with a weak organic base. Here the results are less striking; the lowering of the point of deposition of the salt below the fusing-point of the free base is much more considerable than in the preceding case. However, it is only $1-3^{\circ}$ for hydrochlorate of hexadecylamine. M. Krafft proposes the name of *globomorphous* in stead of amorphous, to indicate the state of the colloidal solutions solidified without crystallisation, admitting that the molecules in such solutions are animated by movements of rotation on themselves, and that they often deposit globular aggregations on cooling.—*Berichte*, xxvii., p. 1596.

MEETINGS FOR THE WEEK.

THURSDAY, 15th.—Chemical, 8. "The Bases contained in Scottish Shale Oil," by F. C. Garrett, M.Sc., and Jas. Smythe, B.Sc., Ph.D.

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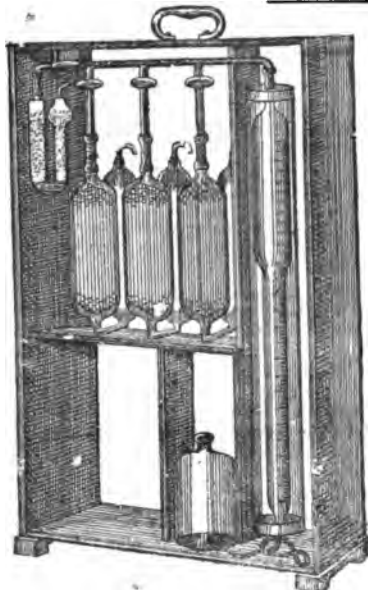
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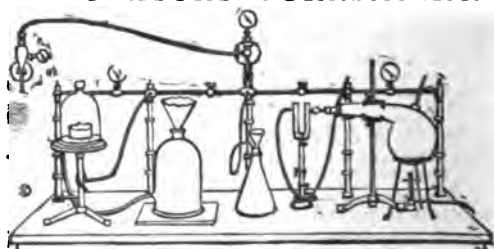
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THE MUTUAL RELATIONS OF IRON, PHOSPHORUS, AND CARBON, WHEN TOGETHER IN CAST-IRON AND STEEL.*

By J. E. STEAD, F.I.C., F.C.S.

(Concluded from p. 223).

ON THE INFLUENCE OF CARBON WHEN MELTED WITH IRON CONTAINING PHOSPHORUS.

WHEN saturated solid solutions of phosphide and iron are melted with increasing quantities of carbon, the effect is very pronounced at first, but comparatively slow after 1 per cent of phosphorus as phosphide has been thrown out of solution. This is shown by the following table:—

Nos.	Carbon. Per cent.	Phosphorus.		
		In free Fe_3P . Per cent.	In solution. Per cent.	Total. Per cent.
1.	nil	nil	1.75	1.75
2.	0.125	0.18	1.37	1.55
3.	0.180	0.59	1.18	1.77
4.	0.70	1.00	0.75	1.75
5.	0.80	1.06	0.70	1.76
6.	1.40	1.16	0.60	1.76
7.	2.00	1.18	0.55	1.73
8.	3.50	1.40	0.31	1.71

That the first portion of phosphorus should be easily removed is not surprising, for, as has been shown, on very slowly cooling a portion of the same iron free from carbon, nearly half of the phosphorus separated without any internal pressure from a third element. When 1 per cent of carbon is present, only 0.70 per cent of phosphorus remains in solid solution and 1.05 separates. With 3.5 per cent carbon this is reduced to 0.31 per cent.

An interesting instance of the effect of carbon in throwing out phosphide was found in a "bear," consisting of partially dephosphorised metal which had remained in the bed of a basic-lined open hearth steel furnace, when it was cooled down for re-lining. The analysis of this metal was as follows:—

Carbon	1.230 per cent
Manganese	0.45 "
Silicon	0.06 "
Sulphur	0.018 "
Phosphorus	1.38 "
Phosphorus in solid solution ..	0.62 "
Phosphorus in free phosphide ..	0.76 "

The microscope revealed the presence of large crystal-line grains of pearlite, in which were embedded perfectly formed idiomorphic crystals of phosphide of iron, globules of phosphide eutectic, and irregularly-shaped masses of carbide of iron. The grains were surrounded by compound envelopes of carbide and phosphide of iron, the phosphide of iron being enclosed in the carbide.

Mr. E. H. Saniter, who primarily examined this specimen, found that, by crushing to fine powder and sieving off the finest part of the crushings, he was enabled to get the pearlite grains in a separate state. These he found, on analysis, to contain 0.62 per cent of phosphorus, a figure corresponding identically with that yielded by the chemical method in the laboratory of the writer; a figure which also corresponds very closely with the results given in Nos. 5 and 6 in Table II. These figures lead us to the conclusion that martensite, in presence of an excess of

phosphide of iron, will dissolve and retain in solid solution between 0.6 and 0.7 per cent phosphorus as phosphide of iron.

The continuity of the phosphide areas in the sample was very remarkable. On heating a portion of the metal in contact with the absorbent material oxide of iron, at a temperature of between 900° and 1000° C., a large portion of the eutectic simply ran out of the mass, and was absorbed in the surrounding oxide of iron. The analyses of the metal before and after treatment are as follows:—

	Before. Per cent.	After. Per cent.
Phosphorus	1.38	0.91
Carbon	1.23	traces
P in Fe_3P (free)	0.76	0.18
Do. (solution)	0.62	0.73
Phosphorus lost—about ..	—	0.47

The phosphide of iron had evidently combined with a portion of the carbide of iron and the surrounding iron to form a fusible eutectic. This, soon after it became liquid, escaped or liquated from the mass, the grains falling together and uniting perfectly. The oxide of iron, on long-continued heating, eventually removed the carbon.

At one time during the heating each grain must have been practically floating in a fluid envelope.

In reflecting over these results it appeared probable that, if several pieces of sheet iron were placed one on the top of each other, and between each a little phosphide of iron eutectic in powder was placed, on heating to about 1000° C. they would all become welded together in the same way as the grains, and the excess of the phosphide would run out at the sides. A practical trial demonstrated that this actually took place. All the pieces were perfectly welded together, and, excepting at the points where the surfaces of the metals were not quite flat, no free phosphide remained.

On heating a sample containing a much larger quantity of phosphorus than 1.3 per cent, in the same way, but in which the phosphide areas were not continuous, no liquation occurred, excepting where the end of the eutectic areas terminated at the exterior of the piece.

When carbon is melted with iron containing less than the saturation quantity of phosphide, if the carbon is in sufficient quantity a part of the phosphide is thrown out of solution, even although the total amount of phosphorus is small.

The following table indicates the result of adding various quantities of phosphorus to steel containing about 1 per cent carbon:—

Nos.	Carbon. Per cent.	Phosphorus.	
		Per cent.	Phosphorus in free phosphide. Per cent.
1.	0.95	0.037	—
2.	0.96	0.099	0.002
3.	0.95	0.122	0.035
4.	0.96	0.347	0.065
5.	1.02	0.548	0.163

No free phosphide could be detected by the microscope in No. 1. In No. 2 it was doubtful if any was present. No. 3 contained traces. Nos. 4 and 5 contained distinct quantities. No. 5 ingot was very red-short, and broke to pieces with the first blow of the hammer.

The microscope showed that the phosphides did not form perfect and continuous envelopes around the grains, but occurred locally segregated at distances apart, and may be classed as typical with ordinary segregation which occurs more or less constantly with every ingot of steel which passes from the solid to the liquid state.

In all cases where segregation of phosphorus in steel has been studied, that element has been accompanied by carbon; in other words, they segregate together.

The segregates, as a rule, are located in those parts of the ingots or masses of steel which are last to solidify. The degree depends on three conditions, viz., the temperature, the mass, and the rate of cooling. With steel at a

* Read before the British Association (Section B), Bradford Meeting, 1900.

temperature much above its melting-point, and of great mass, when it is cooled very slowly, the segregation reaches a maximum.

Under the most favourable conditions there is generally some slight segregation.

It may be admitted that, in all cases when steel solidifies, there is a part which contains more carbon and phosphorus than the rest, and which is the last to solidify. This residuum, although not a eutectic, behaves very like one. It is a kind of mother-liquor. Its final position must depend on the way the greater mass of steel falls out of solution.

If the initial solidification commences at many centres the segregate must take the form of fluid, and finally solid cells, surrounding the parts which first solidified. So far as analysis could detect, in such a case there would be no segregation in the ordinary sense of the term, but it must exist nevertheless, distributed more or less evenly over the whole metal.

On the other hand, when the solidification commences at a few points and steadily progresses from those centres through comparatively great distances, a residuum, high in phosphorus and carbon, must be driven forward in front of the growing crystals till met by those growing in the other direction. When advance is no longer possible it finally solidifies, and forms the comparatively large segregates, easily detected by chemical analysis.

There can be no doubt that in all steels the part which first falls out of solution contains less carbon and phosphorus than that which finally solidifies. It might be urged that if that was so, the microscope would show at once the segregated carbon areas. It actually does so when it is very pronounced in the centre of large ingots, and in small ingots when the carbon is above 0.9 per cent, but not in small masses when the carbon is under 0.8 per cent, for the simple reason that after the residuum has solidified the carbon diffuses out of the areas and passes into the surrounding metal. The phosphides which diffuse very slowly must of necessity be left behind, and these can be sometimes detected by the microscope in high carbon steels, as has been shown. Phosphide of iron eutectic dissolves carbide of iron, but the carbide separates from it at its solidifying point. If then the solidifying phosphide and carbide are surrounded by a mass of iron capable of absorbing the liberated carbide, it is easy to understand how it happens that it is not found segregated with the phosphide.

The peculiar fringe of pearlite containing cementite plates radiating from the phosphide eutectic, in metals containing 1.75 per cent P, 0.12 per cent C, appears to confirm the conclusion that the carbide first segregated with the phosphide as a liquid, and that the carbon is thrown out at the point of solidification or afterwards in cooling down.

ON THE EFFECT OF INTRODUCING SOLID CARBON INTO SOLID PHOSPHORETIC IRON BY THE CEMENTATION PROCESS.

Samples containing variable quantities of phosphorus were carburised in a cementation furnace, at Sheffield, by Mr. T. W. Sorby. The results obtained were all of a very instructive character. It was found that, unless the amount of carbon introduced exceeds 1.20 per cent, no phosphorus is thrown out of solution, until the original phosphorus present exceeds 0.6 per cent, and if it is above that quantity the excess is thrown off as a liquid eutectic which liquates out of the mass, leaving the grains with from 0.6 per cent to 0.7 per cent phosphorus in solid solution.

A small sample, which originally contained 0.88 per cent phosphorus on carburising to 1.20 per cent carbon, contained when it left the carbon furnace only 0.63 per cent phosphorus, and only traces of free phosphide, the difference of 0.25 per cent having been expelled from solid solution by the carbon, and escaped entirely from the metal,

A larger piece of metal, containing about 1.96 per cent phosphorus, yielded a most remarkable result. It was of a very irregular shape, and about 6 c.m. in diameter at the thickest part. When it came out of the cementation furnace it was much scored on the outside, as if fluid metal had been run over it in thin streams. The lower part had adhering to it a solidified drop of metal, which had evidently liquated from the specimen. It was detached for analysis. On fracturing the metal a most instructive structure was revealed.

The metal before treatment was coarsely crystalline, with cleavage faces 3 c.m. across. After carburisation, excepting in a small kernel where the metal had escaped carburisation and where the original structure remained intact, the metal was entirely altered.

The external parts of the structure were coarsely granular, but between these and the kernel the crystalline masses were arranged in columns.

Portions were taken from fixed positions, and these were separately analysed with the following results:—

Description.	Carbon (per cent).		Phosphorus (per cent).		
	Carbon.	Total.	As free Fe ₃ P.	In solid solution.	Lost.
Kernel, centre ..	traces	1.96	0.94	1.02	none
Kernel, average.	0.23	1.94	0.94	1.00	none
1.	0.89	1.52	0.86	0.66	0.42
2.	0.93	1.30	0.61	0.69	0.64
3.	1.20	1.05	0.38	0.67	0.89
4. External layer	1.31	0.91	0.29	0.62	1.03

The drop of metal from the bottom of the piece contained:—

Phosphorus 4.86 per cent

The evidence is conclusive that, as the carbon passed into the metal, its dominating influence threw out the phosphide, equivalent to all excepting about 0.65 per cent.

MICROSTRUCTURE OF CEMENTED METAL.

A section of the metal revealed a most instructive structure when examined under the microscope. The central part, where there was no carbon, was traversed and cut up by plates or prisms of free phosphide of iron, and resembled the same metal after it left the malleable casting-furnace, a coincidence which removes the supposition that oxidising influences were responsible for the reorganisation of the phosphide from the condition it existed in in the metal before treatment. Near to the junction between the kernel and the columnar grains, the eutectic was bordered with a beautiful fringe of pearlite. The cementite laminae radiated from the junction towards the centre, the base of which rested upon a layer of the phospho-pearlite eutectic. The kernel was more or less spherical, and was surrounded by a shell of the same eutectic. This brittle envelope made it easy to break away the surrounding metal, leaving the kernel intact. The radiating columnar masses were encased by a layer of the phosphide eutectic, and the latter was surrounded by a tooth-like formation of cementite. The mass of the columns consisted of pearlite in which were embedded oval, elliptical, and globular masses of the eutectic, some of which were encased by cementite. The amount of the phosphide eutectic decreased with the distance from the centre. The columnar structure terminated at a distance of 1½ c.m. from the centre, and was replaced by ordinary polygonal grains, the joints of which contained very little phosphide, but sufficient carbide to completely envelope them. Imprisoned in the centre of these grains were globular masses of eutectic, and at wide distances apart irregular-shaped masses of the same substance were segregated at the junction of 3 grains.

If the specimen carburised had been perfectly globular, and the carburisation regular at all times during the process, there would have been a spherical fluid area between the centre and the outside, dividing a globe of solid metal

n the middle from a solid shell at the exterior, more or less cut up by fluid cells through which the liquid eutectic would have continually flowed, and which would have eventually left the mass of metal and fallen into the charcoal surrounding it. Although the specimen was not globular, the fluid envelopment must have been developed and continual liquidation of the eutectic been effected.

It is difficult to explain the changes in structure without the hypotheses that there exist allotropic conditions of iron.

In a paper by the writer on the crystalline structure of iron, it was for the first time proved that the thermoperturbation at Ar. 3, between 800° and 900° C., was coincident with the complete reorganisation of the crystalline structure. The coarse-grained, brittle, soft iron, on heating to just above Ar. 3, was changed to material with very fine grain and of exceedingly tough character.

Phosphorus has been proved by M. Osmond and others to prevent the thermal change at Ar. 3, and the writer has found that coarse-grained phosphoretic iron on heating through 900° C. is not reorganised in structure. In fact, a rectangular piece of iron, obtained by cleavage from a large crystalline grain of that material, on heating to between 900° and 1000° C., retained its original orientation with cleavages parallel to those of the metal previous to heating. Pure iron, then, consists of β -iron between Ar. 2 and Ar. 3 and of γ -iron above Ar. 3.

When above 1.25 per cent phosphorus is present in solid solution in iron, it is of β -iron between Ar. 2 and above 1000° C. γ -Iron cannot exist in presence of much phosphorus.

It is clear, then, that as carbon passed into the β -iron, expelling phosphorus from solid solution, the allotropic change under the influence of carbon must have been coincidentally effected, accompanied by a change or reorganisation of the crystalline structure.

A similar kind of columnar structure was developed on decarburising a bar of steel containing 0.21 per cent carbon at a temperature just under 800° C. It was at first difficult to account for the growth of the crystals in this peculiar way; but, on reflection, and knowing that carbon lowers the point at which the change from β to γ is effected, and knowing that γ -iron free from carbon cannot exist at a temperature under 800° C., it will be seen at once that the γ -iron existing under the influence of carbon at 800° must have changed its state when the carbon was removed, and as the removal of the carbon took place steadily from the outside towards the interior, the columnar crystals were formed coincidentally with the passage from γ to β condition—exactly the reverse of what must have taken place when the phosphorus was expelled by the introduction of carbon into the phosphoretic β -iron.

Although carbon in entering into iron containing phosphorus throws phosphide out of solid solution, it limits the amount of carbon which can be absorbed by the iron, and when 15.58 per cent phosphorus is present, the Fe_3P will not absorb carbon even by melting it with charcoal at a white heat. The result of repeated trials has shown that the carbon capable of being absorbed and retained by iron varies approximately inversely with the proportion of phosphide. Thus, if 50 per cent phosphide of iron and 50 per cent of free iron are melted together in contact with charcoal, the carbon absorbed will be only about half as much as if no phosphide was present. The following table shows practical results of melting iron and phosphorus with an excess of carbon:—

Analysis of mixture.		Carbon in melted metals. Per cent.	Fracture.
Iron. Per cent.	Phosphorus. Per cent.		
100	none	4.15	White.
96	4.10	3.25	"
93	7.90	2.00	"
87	13.00	0.70	"
83	16.00	nil	"

ON THE ACTION OF HYDRACIDS ON STEELS CONTAINING CARBON AND PHOSPHORUS.

Referring to the peculiar behaviour of hydracids upon solid solutions of phosphorus in iron, it will be remembered that as the phosphorus is more concentrated the less proportion of it is evolved as phosphine gas. The results of experiments made by M. Osmond have shown that the amount of carbon present influences the proportion of phosphorus evolved as phosphine, PH_3 ; and the experiments of the writer have fully confirmed his results. As the carbon is increased, less and less of the phosphorus present is gasified on dissolving the metal in acid. The only explanation of this peculiarity is based on the assumption that the carbon dominates the greater part of the iron and concentrates the phosphorus into the balance, and that, therefore, as the carbon is increased, the residuum of phosphorus and iron becomes a more and more concentrated solid solution of phosphide in the iron, and behaves in the same way when dissolved in acid as solid concentrated solutions which are free from carbon.

DIFFUSION OF SOLID PHOSPHIDE IN SOLID IRON.

It has been long known that carbon or carbide of iron will diffuse into iron, and Sir W. Roberts-Austen has shown that certain metals will diffuse into lead.

Prof. Arnold has proved that phosphide of iron will diffuse into iron at 1000° C. The results of the writer's observations, that solid phosphide falls out of solid solution and assumes the form of idiomorphic crystals, is strong evidence of molecular movement and diffusion of the dissolved substance in the solid solvent.

THE TITRATION OF MERCURY BY SODIUM THIOSULPHATE.*

By JOHN T. NORTON, Jun.

(Concluded from p. 230).

In dealing with the estimation of mercury in the form of mercurous nitrate, the same method of procedure was employed as in the case of mercuric chloride. A solution of mercurous nitrate was prepared by dissolving as much as possible of 20 grms. of the salt in about 200 c.m.³ of water, filtering off the clear liquid and diluting to a definite volume. The standard of the solution was determined by precipitation as metallic mercury by means of the electric current. Contrary to the statement made in Sutton, the brown precipitate of Hg_2S , formed as shown in the equation,—



does not settle and leave a clear supernatant liquid, but the solution remains cloudy, and it is impossible to see any end reaction. Although the conditions of dilution, temperature, and amount of acid present were carefully considered, no arrangement or adjustment of these conditions was found under which satisfactory results could be obtained. Table V. gives the result of experiments.

The errors in experiments 1, 3, 4, 5, 6, 10, 11, 12, and 17 are, proportionally to the amount of material handled, practically the same, and this fact caused me to make a careful revision of all standards; but no mistake could be found. The reaction upon which the process depends requires the formation of Hg_2S , but this mercurous sulphide breaks down immediately into mercuric sulphide and mercury. The latter is probably acted upon by the free nitric acid present to form mercuric nitrate, which in turn is transformed into the compound $2\text{HgS} \cdot \text{Hg}(\text{NO}_3)_2$ by the action of the thiosulphate. At any rate, with an error so large, whatever its source may be, the process is plainly impracticable.

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, 1900, vol. x., No. 5.

TABLE V.

Hg(NO ₃) ₂ taken as Hg.	HNO ₃ (1:3).	Volume at be- ginning.	Temp. in excess.	Na ₂ S ₂ O ₃ H ₂ g(NO ₃) ₂ found as Hg.	Error.
Grm.	C.m.s.	C.m.s.	°C.	Grm.	Grm.
1. 0.0148	none	50	50	4.28	0.0019-
2. 0.0148	none	75	60	4.45	0.0031-
3. 0.2976	none	300	60	12.67	0.0216-
4. 0.1488	none	100	40	2.19	0.0102-
5. 0.1488	none	100	50	6.92	0.0110-
6. 0.1488	none	200	50	0.78	0.0100-
7. 0.0744	none	100	65	0.73	0.0108-
8. 0.0744	1	100	55	0.49	0.0011-
9. 0.0744	2	100	40	1.16	0.0084-
10. 0.0744	1	100	55	1.35	0.0059-
11. 0.0744	1	100	55	0.82	0.0058-
12. 0.0744	1	200	55	1.71	0.0059-
13. 0.0744	4	100	40	1.77	0.0095-
14. 0.0744	5½	100	40	1.34	0.0099-
15. 0.0744	1	100	45	1.71	0.0090-
16. 0.0744	10	100	45	1.55	0.0075-
17. 0.1488	1	100	40	0.73	0.0097-

TABLE VI.

Hg(NO ₃) ₂ taken as Hg.	Volume at be- ginning.	HNO ₃ (1:3).	Temp.	Na ₂ S ₂ O ₃ in excess.	Hg(NO ₃) ₂ found as Hg.	Error.
Grm.	C.m.s.	C.m.s.	°C.	C.m.s.	Grm.	Grm.
1. 0.1167	200	none	60	0.46	0.1384	0.0217+
2. 0.1167	100	1	60	3.63	0.1348	0.0181+
3. 0.1167	100	2	60	0.23	0.1375	0.0208+
4. 0.1167	100	none	60	0.17	0.1360	0.0193+
5. 0.1167	300	none	21	0.12	0.1232	0.0065+
6. 0.1167	300	none	21	0.15	0.1375	0.0208+
7. 0.1167	200	none	21	11.8	0.1461	0.0294+
8. 0.1167	200	20	21	15.97	0.1403	0.0236+
9. 0.1278	200	none	21	5.23	0.1647	0.0369+
10. 0.1278	200	none	21	1.35	0.1653	0.0375+
11. 0.1278	100	none	21	1.43	0.1662	0.0384+
12. 0.0752	200	none	21	2.09	0.0996	0.0244+
13. 0.0255	100	none	21	2.01	0.0280	0.0025+
14. 0.0255	200	5	21	3.44	0.0264	0.0009+
15. 0.0255	200	10	21	0.85	0.0334	0.0079+
16. 0.0255	300	none	21	1.96	0.0264	0.0009+
17. 0.0255	200	none	21	1.9	0.0287	0.0032+
18. 0.0255	200	none	21	1.76	0.0290	0.0035+
19. 0.0639	200	none	60	1.53	0.0831	0.0192+

The third step in Scherer's process deals with the action of sodium thiosulphate on mercuric nitrate. In the following experiments a solution of mercuric nitrate was prepared either by dissolving as far as possible 20 grms. of mercuric nitrate in about 200 c.m.³ of cold water, filtering off the supernatant liquid, and diluting to a definite volume (Expts. 1-8), or by dissolving the salt in strong nitric acid and diluting (Expts. 9-19). The standard of the solution was obtained by precipitation as metallic mercury by means of the electric current. The yellow precipitate, formed according to Scherer's reaction,

$$3\text{Hg}(\text{NO}_3)_2 + 2\text{Na}_2\text{S}_2\text{O}_3 = 2\text{HgS} \cdot \text{Hg}(\text{NO}_3)_2 + 2\text{Na}_2\text{SO}_4 + 2\text{N}_2\text{O}_5,$$

on adding the sodium thiosulphate, settles much better than in the case of either mercuric chloride or mercurous nitrate; but, as the supernatant liquid takes on a permanent yellow colour towards the end of the reaction, it is impossible to see when the thiosulphate produces no further precipitation. On this account, therefore, the same method of procedure was adopted as in the case of the mercuric chloride and mercurous nitrate, i.e., filtration and titration of the excess of sodium thiosulphate with iodine and starch solution. The result of the experiments is shown in Table VI.

These results seem to show the impossibility of obtaining accurate results according to Scherer's method for the

determination of mercuric nitrate by direct titration with sodium thiosulphate. The constant plus error cannot be accounted for on the hypothesis that the nitric acid present decomposes the sodium thiosulphate, for in that case the error would lie in the other direction. It is more probable that the constitution of the compound $2\text{HgS} \cdot \text{Hg}(\text{NO}_3)_2$ is not definite enough to make it the basis for an analytical process.

In conclusion I wish to thank Prof. F. A. Gooch for his kind advice and assistance.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 228).

Dilatometer Experiments.

Experiment 1.—Amorphous selenium in aniline. The following readings were obtained:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
20°15	9	12:30	21°	107	9:25
—	9	12:45	—	125.2	10:40
29.9	32.5	1:15	—	123.8	10:45
—	32.5	2:45	—	120.5	10:52
40.2	56.7	3:40	—	118.5	10:56
—	56.7	3:50	65.5	104.8	11:15
49.8	79	4:20	—	104.3	11:35
—	79	4:50	—	104.2	11:56
60.1	103.2	5:10	59.9	90.8	12:15
—	103.2	5:35	—	90.8	12:40

The material was then slowly cooled to room temperature.

23.2	3.2	2:28	71	116.8	9:42
23.1	3.0	2:40	—	117.0	11:00
22.5	2.0	4:00	80.2	138.3	11:17
69.6	113.6	4:15	—	138.3	11:31
—	113.7	4:33	97	177.3	12:22
70.8	116.5	4:36	—	177.3	12:37
—	116.3	5:00	—	—	—

The first group of values shows that amorphous selenium in aniline shows no immediate change in volume when heated to 60°. (Later experiments have shown that there is a change when amorphous selenium remains in contact with aniline, even at ordinary temperatures; but this goes on comparatively slowly). The second group shows that at 70° a change sets in, which continues, though slowly, when the temperature is lowered to 65°; the apparent checking of the reaction at 60° was probably due to the fact that all the selenium was already transformed. It is important to notice that there was no sign of a tendency to return to the original volume when the temperature was maintained for twenty-five minutes at 60° and then slowly lowered to the temperature of the room. The remaining values show that no further change goes on up to 97°; the slight variations in tenths of a millimetre on the scale are not to be regarded as significant. In this experiment it was noticed that at 40° the selenium had shrunk together to about half of its original volume, and when the temperature had reached 50° it had become almost black and formed a small mass in the bottom of bulb.

Experiment 4.—Amorphous selenium in aniline, using a larger quantity of selenium than in Expt. 1. In this experiment, as in many which follow, the rise in temperature of the bath was slow and continuous, and readings were taken at intervals; not all of these are recorded here, but enough to make it possible to reconstruct the expansion curve; where any change was perceptible, the temperature was held constant for a time at that point. The selenium used in this experiment was first heated to 50°, to facilitate the liberation of air bubbles which adhere to the powder.

* From the *Journal of Physical Chemistry*, vol. iv., No. 6.

Temp.	Scale.	Time.	Temp.	Scale.	Time.
19°8'	10°3	12 : 20	—	90°3	3 : 03
60°1	97°0	12 : 40	Transferred to cold water.		
—	97°2	12 : 43	20°2	3	3 : 09
60	91	3 : 00	—	3	3 : 40

After standing over night :—

17	—2	9 : 35	66	89	12 : 05
28	18	9 : 50	—	64°5	12 : 50
35	35	10 : 02	—	61	3 : 00
43	52	10 : 15	69	67	3 : 05
48	63	11 : 22	79	87°5	3 : 23
60	87°5	11 : 48	90	109°5	3 : 45
66	95°5	11 : 58	100	129	4 : 15

The dilatometer was then cooled down, and in a subsequent experiment, lasting about four hours, the temperature was slowly raised from 72° to 202° without any irregularity in the expansion curve being observed.

In Experiment 4, the contraction took place in two different stages, separated by a period of cooling and then a slow rise in which the temperature was maintained for one and one-half hours between 43° and 60° without any return toward the original readings. This confirms the evidence of Experiment 1, showing that when both forms of the substance are present, the change is still only capable of going one way, *i.e.*, is irreversible. In a later experiment this dilatometer was heated in direct sunlight during forty-five minutes from 113° to 162°; the readings give no indication of any change in the selenium. It may be as well to state here, what is later established, that the denser form obtained in all these dilatometric experiments is metallic selenium of density 4·8.

It was desirable after completing these experiments to heat the dilatometer up to 220° in order to transform the selenium again into the liquid modification, without being under the necessity of removing it from the bulb. It did not prove practicable to do so in aniline, on account of the evolution of gas, which begins in that liquid at temperatures varying from 160° upwards, according to the pressure within the tube. Aniline was therefore abandoned, and a few experiments were tried with glycerin; the same difficulty was encountered with this liquid, too; nevertheless, several sets of observations were made, up to about 200°, with the result that the transformation of vitreous selenium was found to begin at about 75°, no further change being observable up to 200°, nor was there any sign of a reverse transformation when the tube stood at 25°. The experiments of Siemens and others on the conductivity of selenium indicated the possibility of some change taking place in sunlight at 160° or above, but although the tube which contained metallic selenium was kept for half an hour under a burning-glass, in a bath whose temperature varied between 156° and 172°, no change was found to occur.

Some experiments were then made in quinoline. Starting with metallic selenium, the dilatometer was heated in the dark for five hours between 154° and 196° without any indication of a change at any point. Subsequent heating for three hours between 150° and 200° only confirmed this result. The tube was then brought into direct sunlight, placed under a burning-glass, and heated, on different days, for one and one-half hours between 150° and 180°, for one and one-half hours from 190° to 195°, and for one-half hour from 198° to 202°; the readings are, within the limits of experimental error, identical with the original observations when the heating was carried on in the dark. These observations make it extremely improbable that metallic selenium suffers any change either in the dark or in direct sunlight, when heated to temperatures between 150° and 200°. It does not of necessity follow that the element will behave in the same way when heated in a liquid capable of dissolving it, as it will when heated by itself; on the other hand, the probability is that any change which goes on in the substance taken by itself, which leads to the production of a more stable form, will take place only the more readily in presence of a solvent.

Thus, it will be shown later on that amorphous selenium, which is capable of existing by itself for an indefinite time without change, at ordinary temperatures, goes over rapidly, in the presence of pyridine and certain other liquids, to the metallic form.

The same difficulties which had been met with in working with aniline and glycerin were once more encountered with quinoline when the temperature was raised above 200°. It was supposed at first that all of these reactions were with the liquids themselves, but it seems more probable from later observations that the difficulty is due to small quantities of water which adhere to the selenium from the method of purification. The dilatometers when opened always had the odour of selenium hydride. Meanwhile it had been found that solid paraffin promised to give better results, and this material was then employed for several sets of observations. It was thus rendered possible to work freely between 20° and 230°, although the results of successive transformations into the metallic form did not always lead back exactly to the same reading, on account of the gradual accumulation in the selenium of gas bubbles, which, under the prevailing high pressures, did not free themselves from the mass. This difficulty was obviated later by opening the tube from time to time to let the bubbles escape.

Experiment 23.—Metallic selenium heated in paraffin from 60° to 210° shows no irregularity. When the temperature is raised to about 218°, the selenium begins to melt and expands; at 220° the melting was complete in half an hour, and on cooling, the vitreous form was produced. The readings are as follows :—

Temp.	Scale.	Time.
60°	30	11 : 55
172	257	12 : 10
200	315	
210	336	
218	360	12 : 15 (fusion begun)
222	412°5	12 : 49 (fusion complete)

the tube was then cooled rapidly—

57°5	57	12 : 55
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The dilatometer was then placed in a bath at 160°, whose temperature was rapidly rising. The level in the capillary rose at first, then rapidly fell, showing a sudden transformation from the vitreous to the metallic modification. The tube then stood in direct sunlight for an hour at about 200°. At the end of the time the reading was 311, almost identical with the above. Thereupon the temperature was raised to about 228°, and the material completely fused, after which the bath temperature was brought down to 200°, in order to determine what change takes place when melted selenium is maintained at that temperature. The readings are as follows :—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
225°	418	3 : 42	200°	351	4 : 24
199	367	3 : 45	—	344	4 : 33
199	363		—	339	4 : 40
198	359	3 : 47	—	335	4 : 48
198	357°5	3 : 49	—	330	4 : 55
198	357°5	3 : 52	—	325	5 : 03
197°5	356	4 : 01	—	322	5 : 15
199	356°5	4 : 10	—	320	5 : 25
199°5	355°5	4 : 15	—	320	5 : 36
200	353°5	4 : 20	—	319	5 : 46

The dilatometer was then cooled to 65°, where the scale reading was 30. This reading, like the final reading at 200°, is somewhat higher than that previously obtained. A few small bubbles of gas were observed in the bulb at the end of the experiment, and it is to them, no doubt, that the variations are to be attributed. This experiment in itself would not be conclusive without the confirmation of later observations; the values are given in detail to indicate the rate at which the change goes on at 200°.

The falling level in the first readings at 190° is not due to a change in the selenium, but to the lag of the instrument, which does not adjust itself at once to the changed bath temperature; a closer inspection shows that the transformation of the selenium did not set in until about half an hour later and was complete about an hour after it commenced. The rate is at first very slow, then increases and remains almost constant until near the end, when it is much retarded.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Extra Meeting, July 5th, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

PROFESSOR OTTO PETTERSSON, of Stockholm, delivered the Nilson Memorial Lecture.

On the motion of Dr. ARMSTRONG, seconded by Prof. DEWAR, a vote of thanks was passed to Prof. Pettersson for his lecture.

The following are abstracts of the papers received during the vacation, and published in the *Transactions*:—

111. "Asymmetric Optically Active Sulphur Compounds; *d*-Methylethylthetine Platinichloride." By W. J. PORE and S. J. PEACHEY.

Although the earlier attempts of the authors to resolve methylethylthetine into optically active components were unsuccessful, they have since succeeded in preparing *d*-methylethylthetine platinichloride. Methylethylthetine bromide was treated with silver *d*-camphorsulphonate. The residue, after evaporation at a low temperature, was dissolved in absolute alcohol and precipitated by the addition of anhydrous ether, and this treatment repeated about forty times, when a sparingly soluble fraction was obtained, which seems to be *d*-methylethylthetine *d*-camphorsulphonate. It melts at 118–120°, and gives $[\alpha]_D = +18.6^\circ$ and $[M]_D = +68.0^\circ$.

The *d*-bromocamphorsulphonate was similarly prepared. When either of these salts is dissolved in absolute alcohol, a little strong hydrochloric acid and an alcoholic solution of platinum chloride added, a yellow, crystalline platinichloride is deposited, having the constitution—



and giving $[\alpha]_D = +4.5^\circ$ and $[M]_D = +30.5^\circ$.

112. "Sulvanite, a New Mineral." By G. A. GOYDER.

This mineral was found in considerable quantity in some ore from a new mine near the Burra in South Australia. It appears to afford the first recorded instance of a sulphide mineral containing vanadium as one of its principal constituents.

The analyses given show that it consists of cuprous sulphovanadate, $3\text{Cu}_2\text{S} \cdot \text{V}_2\text{S}_5$. Its hardness is 3.5, and its specific gravity 4.0.

113. "Estimation of Atmospheric Carbon Dioxide." By JAMES WALKER.

A modification of Pettankof's bottle method is described which gives results accurate to 0.1 vol. carbon dioxide in 10,000 vols. air, when a bottle of 2.6 litres capacity (Winchester quart) is employed. The usual error due to absorption of carbon dioxide during titration is avoided by filtering the residual baryta solution into a known quantity of hydrochloric acid, and titrating back with standard baryta. The filtration takes place under diminished pressure through asbestos contained in a Soxhlet filter tube, atmospheric air being entirely excluded during the process of filtering and washing. No special apparatus is employed, and an estimation occupies only half-an-hour.

114. "On some Periodides of Substituted Oxonium Derivatives." By J. N. COLLIE, F.R.S., and B. D. STEELE, B.Sc.

During the investigation of the salts of tetramethylpyrone, it was noticed that when the solution of the iodide was allowed to evaporate in the air brownish crystals slowly separated, which were nearly insoluble in water. From their properties they seemed to resemble the periodides of the pyridine bases. It was found that when free iodine in potassium iodide solution was added to a solution of the hydriodide of tetramethylpyrone or of dimethylpyrone, an immediate crystalline precipitate was produced.

These periodides have the following constitution: from dimethylpyrone, $\text{C}_7\text{H}_5\text{O}_2\text{HI} \cdot \text{I}_2$; from tetramethylpyrone, $\text{C}_9\text{H}_{11}\text{O}_2\text{HI} \cdot \text{I}_2$. They lose iodine when heated, and are at once converted into di- and tetra-methylpyrone when treated with a solution of soda.

The action of iodine on the barium and sodium salts of dimethylpyrone proceeds in a quite different manner. The chief product is an oxidised iodine compound, $\text{C}_7\text{H}_7\text{IO}_3$, which possesses rather remarkable properties; it is not decomposed when warmed with solutions of soda or sodium ethylate, but gives free iodine at once when warmed with a mineral acid.

115. "Condensation of Phenols with Esters of the Acetylene Series. II. Action of Phenols on Ethyl Phenylpropiolate and Ethyl Acetylenedicarboxylate." By SIEGFRIED RUHEMANN and FRED REDDOW.

The authors describe ethyl- β -m-cresoxycinnamate and the products of its transformation as well as the reaction between phenols and ethyl acetylenedicarboxylate. They find that in the latter case aryl ethers of ethyl hydroxyfumarate are formed, and not the ethers of ethyl hydroxymaleate, which were expected; this has been proved by a comparison of the products with the compounds obtained by the action of the sodiophenolates on ethyl chlorofumarate. The aryl ethers of ethyl hydroxyfumarate, on hydrolysis with alcoholic potash, give the corresponding acids, which on heating are partially transformed into the aryl ethers of hydroxymaleic acid; these differ from their stereoisomerides in that lead acetate gives with their aqueous solutions precipitates of their lead salts, but not with the aryl ethers of hydroxyfumaric acid.

116. "The Vapour Pressures, Specific Volumes, and Critical Constants of Di-isopropyl and Di-isobutyl." By SYDNEY YOUNG, D.Sc., F.R.S., and EMILY C. FORTEY, B.Sc.

The results here given, taken in conjunction with those of previous researches, show that an iso- or di-iso-paraffin bears very much the same relation to the isomeric normal paraffin as a lower does to a higher homologue. Thus the ratios of the absolute temperatures to the absolute critical temperatures, and of the volumes of saturated vapour to the critical volumes at corresponding pressures, as well as of the actual to the theoretical densities at the critical points, are higher for the normal paraffins, whilst the ratios of the volumes of liquid to the critical volumes at corresponding pressures are lower.

It was found that di-isopropyl shows marked peculiarities; it is exceptionally difficult to prepare by any method involving the combination of two $(\text{CH}_3)_2\text{CH}$ groups; the specific gravity at 0° and the critical pressure are higher than those of normal hexane, whereas these constants in all other known cases are lower for the iso- and di-isoparaffin than for the normal isomerides; the critical density, the critical temperature, and the boiling-point are also exceptionally high.

117. "The Vapour Pressures, Specific Volumes, and Critical Constants of Normal Octane." By SYDNEY YOUNG, D.Sc., F.R.S.

The normal octane was obtained from Kahlbaum, and was easily purified by treatment with sulphuric and nitric acids and subsequent fractional distillation.

A comparison of the data for normal pentane, hexane,

heptane, and octane shows that the ratios of the absolute temperatures at any series of corresponding pressures to the absolute critical temperatures, and also the ratios of the actual to the theoretical densities at the critical points, increase slightly with rise of molecular weight, whilst the ratios of the volumes of liquid at corresponding pressures to the critical volumes diminish slightly.

The ratios of the volumes of saturated vapour to the critical volumes are, on the whole, very slightly lower than for normal heptane, but are higher than for hexane or pentane.

118. "Separation of Neobornylamine from Bornylamine." By M. O. FORSTER and J. HART-SMITH, A.R.C.S.

In the course of attempts to separate neobornylamine from bornylamine, in addition to the hydrobromide, hydriodide, nitrate, sulphate, and benzoate of bornylamine, the following substances have been obtained.

Camphoroximeacetic acid, $C_{10}H_{16}NO \cdot CH_2 \cdot CO_2H$, which melts at $100-102^\circ$, and gives $[\alpha]_D = -5.9^\circ$; the sodium and bornylamine salts are well defined.

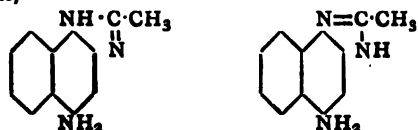
Bornylamine, $C_{10}H_{17}NH \cdot CO \cdot CO \cdot NH_2$, melting at 162° , which gives $[\alpha]_D = -24.1^\circ$.

Dibornylamine, $C_{10}H_{17}NH \cdot CO \cdot CO \cdot NH \cdot C_{10}H_{17}$, which melts somewhat indefinitely at 140° , and gives $[\alpha]_D = -29.6^\circ$.

Purified neobornylamine melts at 184° , and has $[\alpha]_D = -43.7^\circ$ in absolute alcohol. The benzylidene derivative is an oil which boils at 180° under 25 m.m. pressure.

119. "Aminoamidines of the Naphthalene Series." By RAPHAEL MELDOLA, F.R.S., and LEWIS EYNON, A.I.C.

It has been found by the authors that when dinitro- α -acetnaphthalide is reduced by iron and hydrochloric acid (Markfeldt, *Ber.*, 1898, xxxi., 1174), the ethenyltriamino-naphthalene produced is isomeric and not identical with that obtained when tin and hydrochloric acid are used (Meldola and Streatfeild, *Trans.*, 1887, li., 691). In this paper it is shown that the isomerism is entirely attributable to the reducing agent, since the same dinitro- α -acetnaphthalide has been used throughout. Descriptions and analyses are given of Markfeldt's base and some of its salts, of the acetyl derivatives of both bases and their salts, and of the isomeric phenylazo-derivatives. The pronounced difference in the properties of the latter compounds first indicated the isomerism of the respective anhydro-bases. It is suggested that the isomerism may be explained by the structural differences indicated by the formulæ,—



Experiments with the object of determining the constitution of the amino-amidines have been commenced, and the research will be extended in this direction. When the NH_2 group in Markfeldt's base is replaced by hydrogen by the diazo-method, an ethyldiaminonaphthalene is obtained which is apparently identical with that of Prager (*Ber.*, 1885, xviii., 2161).

120. "Note on the Elimination of a Nitro-Group during Diazotisation." By RAPHAEL MELDOLA, F.R.S., and ELKAN WECHSLER.

When acetorthoanisidine is nitrated with excess of fuming nitric acid in the cold in acetic acid solution, a dinitroacetanisidine is formed. This compound crystallises in light yellow needles, melting at $162-163^\circ$, and on hydrolysis by alcoholic sodium hydroxide gives a dinitroanisidine, crystallising in bright orange needles, having a melting-point of $186-188^\circ$. This dinitroanisidine, on treatment with sodium nitrite and an acid, forms a diazo-compound which crystallises in ochreous scales or yellow

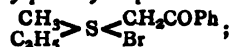
needles, having an exploding-point of about 178° . The diazo-compound has the formula—



so that one nitro-group is eliminated during the process of diazotisation. The corresponding iodonitromethyl-resorcinol crystallises in flat, yellow needles, melting at $115-116^\circ$. The nitroazo- β -naphthol derivative, obtained by combining the diazo-compound with β -naphthol in alkaline solution, forms glittering, bronzy-green scales decomposing at about 250° . The constitution of the new dinitroanisidine is being investigated, with the object of determining the particular configuration which is favourable to this easy displacement of a nitro-group.

121. "A Contribution to the Stereochemistry of Sulphur; an Optically Active Sulphine Base." By S. SMILES, B.Sc.

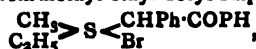
Methyl ethyl sulphide unites with ω -bromacetophenone to form methylalylphenacylsulphine bromide,—



this, when treated in alcoholic solution with silver dextro- α -bromocamphorsulphonate, gives rise to two salts, the molecular rotation of the less soluble of which is $+250^\circ$, and of the more soluble $+289^\circ$. This points to the fact that the basic methyl ethyl phenacyl radicle possesses a molecular rotation in aqueous solution of over 20° .

From the less soluble salt a levorotatory sulphine picrate was obtained as yellow needles (m. p. 125°) for which $[\alpha]_D = -9.3^\circ$ and $[M]_D = -39.3^\circ$. The dextro-rotatory picrate obtained from the more soluble salt (yellow needles, m. p. $123-124^\circ$) gave $[\alpha]_D = +8.1^\circ$ and $[M] = +34.2$.

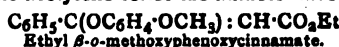
It was not found possible, however, to separate two stereoisomers from methyl-ethyl-desyl-sulphine bromide,—



which was obtained as a colourless mass of deliquescent needles.

122. "Condensation of Phenols with Esters of the Acetylene Series. Synthesis of Benzo- γ -Pyrones." By S. RUHEMANN and H. E. STAPLETON.

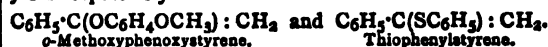
By the condensation of guaiacol and thiophenol with esters of the acetylene series the authors have obtained—



and—

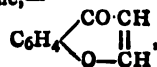


These esters, on hydrolysis with alcoholic potash, give the corresponding acids which lose carbon dioxide and yield respectively—



Ethyl acetylenedicarboxylate condenses with thiophenol to ethyl thiophenylfumarate, which is decomposed by alcoholic potash, forming phenyl disulphide ($C_6H_5S_2$). Along with the ethyl thiophenylfumarate, ethyl dithiophenylsuccinate is also formed, having probably the formula $CO_2Et \cdot C(SC_6H_5)_2 \cdot CH_2 \cdot CO_2Et$. The analogous oxygen compound, along with ethyl β -phenoxyfumarate, is obtained by the action of phenol on ethyl acetylenedicarboxylate.

Ethyl β -phenoxyacrylate, by treatment with strong sulphuric acid, is not condensed into flavone, but decomposes into benzoylactic acid along with oxalic and acetic acids. Ethyl phenoxyfumarate, however, partially undergoes the desired change, forming benzo- γ -pyrone-carboxylic acid, which, on heating, loses carbon dioxide, giving benzo- γ -pyrone,—



melting at 59° .

123. "Contributions to the Chemistry of Hydrotetrazines and Triazoles." By OSWALD SILBERMAD, Ph.D.
The action of hydrazine hydrate on diacetanilide gives dimethyldihydrotetrazine,—

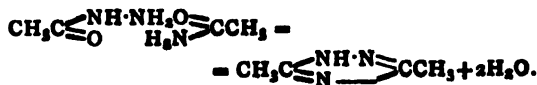


its hydrochloride melts at 232°. Benzoyl chloride decomposes it with the formation of *sym*-dibenzoyl-hydrazine and acetic acid.

Nitrous acid converts it into dimethyltriazole nitrate (m. p. 125°), from which the hydrochloride (m. p. 199°) and the free base,—



(m. p. 142°, b. p. 258° at 752 m.m.), may be obtained. Its constitution is evident from its synthesis from acetamide and acetylhydrazine,—



Diphenyldihydrotetrazine,—



may be obtained in almost theoretical quantity by heating benzoylhydrazine with hydrazine hydrate to 230°. It melts at 264°; Finner gives 258° (*Ber.*, 1894, xxvii., 1006).

Heated alone, benzoylhydrazine yields only traces of diphenyldihydrotetrazine, the chief products being diphenyltriazole,—



and diphenyldiazoxole,—



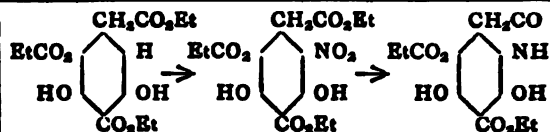
α -Benzoylphenylhydrazine under similar treatment does not condense, but breaks down with production of benzophenone, benzoic acid, and benzoylanilide.

124. "Isomeric Dibenzylketone Benzalanilines and Deoxybenzoin Benzalanilines." By FRANCIS E. FRANCIS, Ph.D., B.Sc.

Three isomeric addition products of the two ketones, dibenzyl ketone and deoxybenzoin, with benzalaniline are described. For the sake of convenience these are termed α -, β -, γ -modifications. The α is obtained from the pure ketone, the β by the action of traces of piperidine on the α , or with greater difficulty on the γ , the γ by the action of traces of sodium ethylate on either the α or β . By the action of heat, the β and γ are transformed into the α -modification. The dibenzyl ketone benzalanilines are more basic than the deoxybenzoin benzalanilines; the former yield normal, whilst the latter give basic hydrochlorides. These salts are different in the case of α and β , but identical in β and γ . They are dissociated by water, giving the corresponding base, but with alcohol the α -hydrochloride appears to give a mixture of α and γ -base, whereas the β or γ gives the α -modification nearly pure.

125. "Condensation of Methyl Acetonedicarboxylate. Constitution of Orcinoltricarboxylic Esters." By F. W. DORTON, M.A.

It is shown that a moderate temperature is sufficient to condense methyl acetonedicarboxylate to trimethyl orcinoltricarboxylate, and that a good yield of the latter is obtained. The constitution assigned to ethyl orcinoltricarboxylate by Jordan is confirmed, and the similarity of the methyl and ethyl esters is shown by nitration and subsequent reduction, when a lactam structure results in both cases, proving that in orcinoltricarboxylic esters the remaining hydrogen atom of the benzene nucleus is in the ortho-position to the side chain, thus:—



126. "Contribution to the Chemistry of the Aromatic Meta-diamines." By G. F. MORGAN, D.Sc.

In this paper the following compounds are described:—1-Bromo-2:4-phenylenediamine melting at 111–112°; its dibenzoyl and its diacetyl derivatives melting at 178.5° and 190° respectively; dibenzoyl 1-chloro-2:4-phenylenediamine, m. p. 178°, and chlorochrysoidine, $\text{PhN}_2\text{C}_6\text{H}_4\text{Cl}(\text{NH}_2)_2$, m. p. 149°; *m*-phenylenediacetyldichloramine, $\text{C}_6\text{H}_4(\text{NCl}\cdot\text{Ac})_2$, m. p. 150–151°; diacetyl 1:5-dichloro-2:4-phenylenediamine, m. p. above 260°; 1:5-dichloro-2:4-phenylenediamine melting at 136–137° and its dibenzoyl derivative at 187°.

Diacetyl-*m*-tolulylenediamine, when chlorinated and then hydrolysed, gives 5-chloro-2:4-tolulylenediamine, m. p. 120–121°; its diacetyl derivative melts at 239–240°.

127. "Action of Aromatic Aldehydes on Derivatives of β -Naphthylamine." By GILBERT THOMAS MORGAN, D.Sc.

The compound, $\text{C}_{10}\text{H}_7\text{N}_2\text{O}_2$, produced by the condensation of ethyl β -naphthylamine (2 mols.) with benzaldehyde (3 mols.) crystallises in colourless leaflets and melts at 148°. Its constitution may be represented by one of the following formulae:— $\text{Ph}\cdot\text{CH}(\text{NEt}\cdot\text{C}_{10}\text{H}_7)_2$, $2\text{Ph}\cdot\text{COH}$;



Its general behaviour is in accordance with the third of these; but freezing-point determinations seem to indicate that the substance, dissolved in benzene, corresponds with the second formula.

The following anhydro-bases and their hydrocyanides, derived from 1-chloro-2-naphthylamine and its bromine analogue, are also described in the paper:—Benzylidene-1-bromo-2-naphthylamine, m. p. 93–94°, and its hydrocyanide, m. p. 92°; the cuminyldene derivative and its hydrocyanide, melting at 100–101° and 120° respectively; the *p*-hydroxybenzylidene, and *o*-hydroxybenzylidene derivatives, melting respectively at 189–190° and 144°, and the corresponding hydrocyanides at 144° and 152°; *p*-methoxybenzylidene-1-bromo-2-naphthylamine and its hydrocyanide, melting at 107° and 140–143°; cinnamylidene-1-bromo-2-naphthylamine, m. p. 126°, and its hydrocyanide, m. p. 142–143°; benzylidene-1-chloro-2-naphthylamine and the corresponding cuminyldene derivative, melting respectively at 99° and 85°, and their hydrocyanides at 77° and 117°; cinnamylidene-1-chloro-2-naphthylamine, m. p. 134°, and its hydrocyanide, m. p. 155–156°; *p*-hydroxybenzylidene-1-chloro-2-naphthylamine and the corresponding *ortho*-isomeride, melting respectively at 191° and 153°, and their hydrocyanides at 152° and 148°; *p*-methoxybenzylidene-1-chloro-2-naphthylamine, m. p. 117°, and its hydrocyanide, m. p. 132°.

The following anhydro-bases do not combine with hydrogen cyanide:—*o*-Nitrobenzylidene-1-chloro-2-naphthylamine, m. p. 142°, and the corresponding bromo-compound, melting at 138°; *p*-nitrobenzylidene-1-chloro-2-naphthylamine, m. p. 151°, and its bromine analogue, melting at 154–155°.

128. "Action of Hydrogen Peroxide on Carbohydrates in the presence of Ferrous Salts. II." By R. S. MORRELL, M.A., Ph.D., and J. M. CROFT, M.A., B.Sc.

Rhamnose when oxidised by hydrogen peroxide in the presence of ferrous sulphate yields an osazone which reacts with phenylhydrazine at the ordinary temperature to give rhamnososazone.

Similarly, cane sugar, when the hydrogen peroxide is neutral, is first "inverted" and then oxidised, since, on testing with phenylhydrazine, glucosazone is precipitated. The action of potassium persulphate on glucose in the

presence of ferrous sulphate is slow at ordinary temperatures. On warming to 40° , oxidation proceeds more rapidly with formation of glucosone. The yield of the glucosone (measured by the weight of the glucosazone precipitate) is smaller than when hydrogen peroxide is employed as the oxidising agent.

129. "*The Specific Gravities of the Halogens at their Boiling-points, and of Oxygen and Nitrogen.*" By J. DRUGMAN, Ph.D., and W. RAMSAY, F.R.S.

The determinations made by the authors of the specific gravities of the halogens at their respective boiling-points under atmospheric pressure have given the following results:—

Iodine boiling at 184.5°	has the sp. gr.	3.706
Chlorine " -33.6°	" "	1.507
Fluorine " -187°	" "	1.108

The specific gravity of fluorine was arrived at by using the data of Moissan and Dewar (*Proc.*, 1897, xiii., 180) and calculating the specific gravity at its boiling-point, corrections being made for the expansion of the liquid fluorine and the change in the specific gravity of amber. Amber was found to have the specific gravity 1.065 at 15° and 1.10 at -187° . The specific gravities of oxygen and nitrogen were found to be 1.315 and 0.7914 at their respective boiling-points.

130. "*On Hydroferrocyanic Acid.*" By K. C. BROWNING, B.A.

The purification and some of the properties of hydroferrocyanic acid are described. This acid begins to evolve hydrocyanic acid at 120° , and its decomposition is complete at 300° , ferrous cyanide being left as a pale yellow powder. Ferrous cyanide decomposes above 430° into iron, carbon, and iron carbide. Reasons are given for considering it to be an isocyanide, but when strongly heated it seems to decompose partly as if it were a normal cyanide.

From the decomposition of ethyl ferrocyanide it would seem that all the cyanogen groups present have the isocyanide arrangement.

131. "*On the Nature of Metal-ammonia Compounds in Aqueous Solutions.*" Part I. By H. M. DAWSON and J. McCRAE.

The nature of various ammoniacal salt solutions has been studied by determining the amount of ammonia extracted by shaking a known volume of the aqueous solution with a known volume of chloroform. If pure aqueous ammonia be shaken with chloroform at a constant temperature till equilibrium is established, this condition is characterised by the relationship $c_1/c_2 = \text{constant}$ (26.3), where c_1 = concentration of ammonia in water and c_2 that in chloroform.

When this is done with ammoniacal solutions of cupric sulphate, cupric chloride, zinc sulphate, cadmium iodide, and nickel sulphate, the constant is found to give much higher values, and hence the amount in combination with the salt may readily be deduced. Experiments with calcium chloride solution also indicate the formation of a complex metal-ammonia compound, but the amount of ammonia combined per molecule of salt is very much smaller than in the case of the other salts investigated.

Experiments were also made with ammoniacal copper oxide solution which indicate that the base $\text{Cu}(\text{NH}_3)_2(\text{OH})_2$ is probably formed; the copper sulphate compound has the formula $\text{Cu}(\text{NH}_3)_4\text{SO}_4$.

PHYSICAL SOCIETY.

Ordinary Meeting, November 9th, 1900.

Prof. A. W. REINOLD, F.R.S., Vice-President, in the Chair.

DR. R. A. LEHFELDT read a paper on "*Electromotive Forces and Osmotic Pressure.*"

This paper is an attempt to explain a difficulty in the interpretation of the ordinary logarithmic formula for the E.M.F. between a metal and solution, pointed out by the author at the Dover meeting of the British Association. An expression for the E.M.F. of a concentration cell is obtained thermodynamically upon the assumption that the electrolyte is only partially dissociated. A partition is used which is permeable to water, but not to the salt or its ions, and the conclusion follows that the E.M.F. depends not on the osmotic pressure of the metallic ions, but on that of the solution as a whole. A graphical representation is given plotting osmotic pressure against dilution, assuming Boyle's law to hold, and it is shown that the E.M.F. is not proportional to the integral $\int PdV$, but to the converse integral $\int VdP$. Assuming,

further, that the osmotic pressure changes according to Van der Waals's equation, the E.M.F. is greater than that calculated from Boyle's law. If the electrolytic solution pressure is calculated from the integral $\int PdV$ it comes

out 10^{11} atmospheres, but if from the converse integral the value obtained is about 20,000 atmospheres. A comparison between actual E.M.F.'s and those derived from the equation given by the author should afford, if the formula is correctly deduced from the assumptions made, a measure of how far the osmotic pressure deviates from that indicated by Boyle's law. Experiments upon concentration cells have been made by Helmholtz, Wright and Thomson, Moser, Lussana and Goodwin, but as their work was performed upon cells with migration of ions, the calculation of the osmotic pressure is rendered uncertain by the introduction of the transference ratio. Accordingly the author has measured the E.M.F.'s of cells without migration, using zinc as electrodes, and chloride and sulphate of zinc as salts. The E.M.F. was measured by the compensation method, using a post office box through which a current was sent by an accumulator. The accumulator kept up a constant potential difference, and was standardised daily by means of a Clarke cell. The experimental results agree with the calculated over the range centi- to deci-normal, showing that the deviation from the value given by the logarithmic formula is accounted for by the incomplete dissociation of the salts. The osmotic pressures are then calculated from the E.M.F.'s, and the values of PV plotted. They show irregularities due to the combined effect of the decreasing dissociation of the salt, and the increasing departure from Boyle's law. Dividing the product PV by van't Hoff's factor, determined from conductivity, values are obtained showing variations similar to those observed in the behaviour of gases when subjected to high pressure.

MR. WHETHAM said there was one form of membrane which is quite permeable to water, and yet does not allow either salts or the ions to get through. He referred to the free surface of the solution itself. The water being volatile can get out, but the salt cannot.

DR. DONNAN said the author seemed to have discovered things well known; for instance, the integral $\int VdP$ is generally taken as proportional to E.M.F. He expressed his interest in the explanation of the difficulty in the logarithmic formula.

DR. LEHFELDT, in reply, said Goodwin had used the integral $\int VdP$, but had not made any numerical calculations by means of it.

MR. R. J. SÖWTER read a paper on "*Astigmatic Lenses.*"

An astigmatic lens is one which so acts on rays of light falling on it as to produce in general two focal lines in the refracted ray system. A lens derived from a quadric surface is the general elementary type of astigmatic lens, and in the paper an ellipsoidal lens is selected and considered. The focal lines are parallel to the elliptic axes, and

correspond to the lens powers in these directions. These powers are proportional to the inverse squares of the axes. A curve drawn through all points on a lens where the material thickness is constant may be said to determine a natural aperture for that lens. A method of natural apertures is employed to establish the various relations set out in the paper. An ellipse is the natural aperture for an ellipsoidal lens, a circle for a spherical lens, and an infinitely long rectangle for a cylindrical lens. It is shown that two cylindrical lenses crossed at right angles are equivalent to an ellipsoidal lens, and the power of the combination in any direction is the same as that of the ellipsoidal lens in that direction. It is also shown that two obliquely crossed cylindrical lenses are equivalent to an ellipsoidal lens, or to two cylindrical lenses of definite powers crossed at right angles, or to a cylindrical and a spherical lens, for a spherical lens may be replaced by two equal cylindrical lenses crossed at right angles.

Prof. S. P. THOMPSON said he had never seen the treatment of an ellipsoidal lens before, although the extreme case of a paraboloidal lens had been considered. The author's method was, so far as he knew, new, and would be very convenient to work with.

Mr. A. CAMPBELL then read the following papers:—

(a). "On a Phase-turning Apparatus for Use with Electrostatic Voltmeters."

Electrostatic voltmeters are particularly insensitive at the lower parts of their ranges, the divisions closing in very much towards the zero point. When measurements of small direct-current potential differences have to be made, it is an easy matter to add to the voltage to be measured a constant voltage large enough to bring the deflection to an open part of the scale. If the small voltage to be measured is an alternating one, it is necessary that the auxiliary voltage should alternate with the same frequency, and be in phase with it. The apparatus described enables the phase of the auxiliary voltage to be turned until it agrees with the one to be measured. The phase difference referred to is not the time lag, but the angle whose cosine is the power factor, and may be called the power lag. The method is to get two independent equal voltages, U_1 and U_2 , differing in power phase by $\pi/2$, and to add together suitable fractions of these, such as $U_1 \sin \phi$, $U_2 \cos \phi$. The resultant is equal to U_1 , but with the power phase turned through ϕ . The unknown small voltage is connected in series with an auxiliary voltage and a voltmeter, and the phase of the latter voltage is turned until the maximum deflection is obtained.

(b). "On a Method of Measuring Power in Alternating Current Circuits."

The circuit in which the power is to be measured is connected in series across the supply circuit with a small non-inductive resistance. By means of a transformer the small voltage on this resistance may be transformed into one whose power phase is π behind the voltage on the resistance. This is added to the voltage on the circuit to be measured and then reversed, and added again. The difference of the squares of these effective resultants is shown to be equal to a constant into the power to be measured. If there is any direct current it must be measured separately by a Weston voltmeter or other suitable instrument.

(c). "Note on obtaining Alternating Currents and Voltages in the same Phase for Fictitious Loads."

When testing instruments for the measurement of large amounts of electrical power or energy, it is usually desirable to do so by means of fictitious loads, i. e., by applying to the instrument under test current and potential difference representing the required load. In order to obtain a fictitious non-inductive load with alternating currents, the potential difference and current should be in the same phase. The current for the instrument under test is got by means of a transformer worked on a hundred-volt circuit. The potential difference in the same phase is got by allowing the current to flow through a non-inductive

resistance, and increasing the voltage at the ends of the resistance to the required amount by means of another transformer.

The Society then adjourned until November 23rd.

NOTICES OF BOOKS.

Handbook of Spectroscopy. (Handbuch der Spectroscopie).

By H. KAYSER, Professor of Physics at the Bonn University. Vol. I. Leipzig: S. Hirzel. 1900. Pp. 781.

For the last ten years Prof. Kayser has devoted a large portion of his time to the compilation of his great work on Spectroscopy, the first volume of which is now before us. In this volume the author has dealt with the subject up to the beginning of 1899, and there remains a large amount of work to complete his record of all that has been written and done since then up to the present time.

This volume is divided into six chapters, each of which is subdivided into a number of sections. Chapter I. deals generally with the science of spectroscopy, beginning with the work of Newton, Herschel, Wollaston, Fraunhofer, and many other distinguished workers. The subject is so vast that we can only briefly follow the author in his history of the growth of the science; emission and absorption spectra, Brewster's three-colour theory, the theory of heat rays, phosphorescence and fluorescence, the spectra of gases, and spectrophotography are but a few of the important points here fully discussed.

Chapter II. is on the production of luminous vapour, subdivided into sections on flames, the electric arc, and the electric discharge. Chapter III. is an important one, and is devoted to prisms; the theory of the prism is fully gone into, in the first section, and its construction in the second; the comparative advantages of and improvements in various prisms are given, including those made from such substances as fluorite, sylvine, rock salt, quartz, and calc spar. We next come to diffraction gratings, and then, in Chapter V., to the construction of the spectroscope, and of spectroscopic apparatus. In this particular branch of the subject we are almost overwhelmed with the number of workers; nearly every chemist and physicist of note seems to have added his quota to the development of the still-growing instrument, while those who cannot lay claim to any improvements have, on the other hand, made excellent use of the instruments as they stood.

Chapter VI. and last is on spectroscopic measurements, and is divided into two principal parts, viz., absolute measurements and relative measurements. This subject is one of the greatest importance, as by its means the results obtained by different authorities can be compared together, and new results criticised and tabulated.

Prof. Kayser is an acknowledged authority on this subject, and the excellent work he has just given to us will be received with the appreciation it deserves. His numerous publications on the spectra of the elements are widely known and universally accepted as standard works. It is no light matter to undertake so thorough a history of spectroscopy, and though the work done and published up to within a year or two may be looked upon as settled, and not likely to require much reconsideration, the work that is now going on is enough to prevent any book being actually up to date when published, as even while it is passing through the press something new is almost bound to be announced.

Besides a full table of contents, this volume has two good indices, one of author's names and the other for subject matter. It is also embellished with 231 illustrations.

Partial Synthesis of Laudanosine.—Amé Pictet and B. Athanasesco.—Laudanosine, $C_{21}H_{27}NO_4$, one of the least abundant alkaloids contained in opium, was prepared from another opium alkaloid, papaverine, $C_{20}H_{21}NO_4$. This partial synthesis fixes the composition, that of papaverine having been already established by M. Goldschmidt.—*Comptes Rendus*, cxxxi., No. 17.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

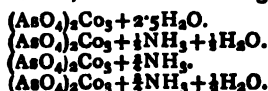
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 17, October 22, 1900.

Diagnoses of Gaseous Supersaturations, of a Chemical and Physical Order.—M. Berthelot.—The author's experiments, which are fully described, establish the fact that oxygen, though susceptible of existing dissolved to a considerable extent in mixtures of hydrogen peroxide and permanganate of potash, really is present in a state of unstable combination. When in the form of hydrogen peroxide, the peroxide is capable of being violently decomposed, with evolution of a considerable quantity of heat; it being, in fact, a chemical supersaturation.

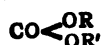
Origin of Atmospheric Hydrogen.—Armand Gautier.—The various phenomena examined by the author seem to point to the fact that the hydrogen present in the air is originally derived from the water: the water reaching on terrestrial substances at a remote period and causing the hydrogen to accumulate. This, however, apparently is only one of the origins of atmospheric hydrogen; the second origin seems to be the residue left in the air, at the moment of the formation of rain water, by the combustion of this hydrogen gas in presence of excess of oxygen, nitrogen, carbonic acid gas, and various vapours.

Index of Refraction and Dispersion of Bromine.—Ch. Riviére.—The physical constants of bromine have been little studied, partly on account of the extreme care necessary in the preparation of pure bromine and also because of the difficulty of manipulation of this substance. Various contradictory numbers have been published. The author examines these numbers afresh, with the aid of improved apparatus. The numbers published show an extraordinary dispersive power; between lines A and D at 20° C. this power is 0.037 as compared with 0.016 for flint and 0.030 for carbon disulphide. The bromine used in these experiments was prepared by the action of sulphuric acid on a mixture of pure bromide and bromate of potassium, then washed with a large quantity of water and dried with phosphoric anhydride.

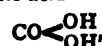
Ammoniacal Arsenates of Cobalt.—O. Dacru.—If a solution of cobalt rich in ammoniacal salts and containing a sufficient proportion of free ammonia, is added to arsenic acid or a double arseniate, a voluminous gelatinous precipitate comes down of a violet-blue colour. If this is kept on the water-bath, the precipitate gradually becomes modified, contracts, and changes to a dark red body, the microscopic examination of which shows it to be completely crystallised. These compounds are the ammoniacal arsenates of cobalt, and it is found that cobalt forms three distinct ammoniacal arsenates, derived from erythrine by the displacement of water by ammonia, molecule for molecule; the four salts being—



A General Method of Preparation of Mixed Carbonic Ethers of the Phenols and the Alcohols.—E. Barral.—The mixed phenolic-alcoholic carbonates—



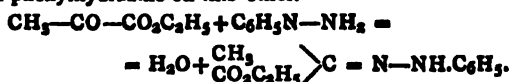
are the ethers of carbonic acid—



in which the atoms of hydrogen have been replaced, one by the phenolic radicle R and the other by the alcoholic

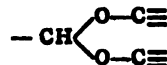
radicle R'. All the rational methods for the preparation of these substances fail, the author showing that all the difficulties arise from the easy formation of the neutral alcoholic and phenolic carbonates, $\text{CO}(\text{OR})_2$ and $\text{CO}(\text{OR}')_2$. He therefore decomposes the chlorocarbonates under the influence of alcoholates or phenates, very unstable bodies, which act as caustic alkalis.

Stereochemistry of Nitrogen. The Stereo-isomeric Hydrazones of Ethyl Pyruvate.—L. J. Simon.—The author succeeds in obtaining under two stereo-isomeric forms the hydrazine of ethyl pyruvate, by the direct action of phenylhydrazine on this ether.

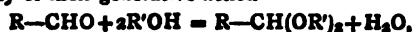


The molecular weight of this substance and the estimation of carbon, hydrogen, and nitrogen, show it to have the above composition.

Acetals of Monovalent Alcohols.—Marcel Delépine.—The author examines several derivative containing the group—



called the formals and the acetals, prepared respectively from formic and acetic anhydrides. He calculates the thermic capacity of their generative action—



in order to establish the nature of the variations in the heats of combustion and of formation by the passage from one form to the other.

Direct Nitration in the Fatty Series.—L. Bouveault and M. Wahl.—By the nitration of ethyl dimethyl acrylate with fuming nitric acid, the author succeeds in preparing a substance of formula $\text{C}_7\text{H}_{11}\text{NO}_4$, named ethyl nitro-dimethyl acrylate. This is a clear yellow liquid, of density 1.1384 and agreeable odour. When the alcoholic solution of this substance is treated with alcoholic potash the potassium salt is formed, $\text{C}_7\text{H}_{10}\text{NO}_4\text{K}$. A solution of this can be decomposed by dilute hydrochloric acid, when a dark heavy oil appears, of formula $\text{C}_7\text{H}_{11}\text{NO}_4$. This compound is isomeric with ethyl dimethyl acrylate. It boils at a lower temperature, 104°—105°, and has density 1.1279, besides being colourless when pure.

Messrs. A. Gallenkamp and Co.—This firm has recently placed on the market an improved form of balance for scientific purposes, of the highest accuracy with constant sensibility. The great drawback in all analytical balances at present in use is, that within the limit of the maximum weight-charge for which they are constructed, they become less and less sensitive with the increase in the charge; the cause of this is in the bending of the beam, by which the end knife-edges are gradually brought below the plane in which all three should be. To eliminate such errors Messrs. Gallenkamp have constructed a new beam, model 1900, the end supports of which are outside the knife-edges; they claim that this construction is all-important, as it gives absolute rigidity to the beam.

Notes on the Estimation of Boric Acid.—C. Montemartini.—Of all the methods proposed for the estimation of boric acid Gooch's method is the only one giving exact results. It consists of liberating the boric acid by means of nitric or acetic acid, and then transforming it into methylic ether. This latter is collected on a weighed quantity of lime, dried, calcined, and re-weighed; the increase of weight is due to the boric acid held. Rosenbladt's method is not a good one, neither is Thadceff's. This latter is very complicated and inconvenient, and does not give anything like such good results as Gooch's method.—*Gazz. Chim. Ital.*, p. 344.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

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MEETINGS FOR THE WEEK.

WEDNESDAY, 21st.—Society of Arts, 8. Opening Address of the 147th Session, by Sir John Evans, K.C.B., F.R.S.
Microscopical, 7.30. Exhibition of Slides illustrating the Structure of Shells.

FRIDAY, 23rd.—Physical, 5. "The Anomalous Dispersion of Carbon," by Prof. R. W. Wood. "The Liquefaction of Hydrogen," by M. W. Traversa. "Refraction of Sound by Wind," by Dr. E. H. Barton.

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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2139.

THE ACTION OF AMMONIA UPON FERROUS CHLORIDE AND BROMIDE.

By GILBERT J. FOWLER, M.Sc.

THE experiments to be described were made in the course of a research on the preparation and properties of iron nitride, and their general results are given in a paper on that compound. The details, however, seemed to be of sufficient interest to publish in a separate note.

Although the action of ammonia upon ferrous chloride was not found to be the best method for preparing nitride of iron, yet a number of experiments were made with the view of gaining a more certain knowledge of the action of ammonia in this case, and to determine especially whether any hydrazine derivatives were formed. The ferrous chloride was prepared by heating iron wire to redness in a current of hydrogen chloride, the crystalline product obtained being freed from intermixed metallic iron by means of a magnet.

In the cold, ammonia is very rapidly absorbed by ferrous chloride, the crystals swelling up to a white powdery mass with evolution of heat, the temperature rising to above 40° C. On introducing ferrous chloride into ammonia gas over mercury, the ammonia is instantaneously absorbed, the experiment being a striking one for lecture illustration. Quantitative experiment showed that from 5 to 6 molecules of ammonia are thus taken up. On exposure to the air the white mass rapidly turns brown, with formation of hydrate of iron. On heating the white substance to the boiling-point of alcohol, in a tube connected with a manometer, no appreciable amount of ammonia was evolved; at 100°, however, a rapid evolution of this gas took place.

In order to investigate the action of ammonia upon ferrous chloride at higher temperatures, the ferrous chloride was spread, in a thin layer, in a hard glass tube contained in a Lothar-Meyer furnace, giving temperatures which can be kept regular up to 500°. Ammonia gas was first of all passed through at the ordinary temperature till all the air was expelled. The temperature was then raised, and at 160° ammonia came off very rapidly from the white compound of ammonia and ferrous chloride, formed in the cold, but no gas unabsorbed by hydrochloric acid was obtained. At 300° this ammonia had not ceased coming off, but no sublimation of ammonium chloride was noticed.

At a temperature above 300°, but below the melting-point of lead, 335°, the mass began to melt and darken, a large amount of ammonium chloride was formed, marking a decomposition of the ferrous chloride, but very little unabsorbed gas was evolved. At the melting-point of zinc, 433°, a small amount of gas was found to be unabsorbed by the acid in the nitrometer connected with the apparatus. On allowing the apparatus to cool, complete absorption of the ammonia took place in the nitrometer, showing that the gas collected was not due to any air having diffused into the apparatus. The temperature was kept low in this experiment, in order better to observe the formation of products intermediate between ferrous chloride and nitride of iron, which, according to Fremy, is the final product of the reaction. The gas evolved was found to be non-inflammable, and therefore was not hydrogen, and since, as mentioned above, it was not air, would seem therefore to consist of nitrogen, produced in the course of the reaction. The solid residue in the tube consisted partly of unaltered ferrous chloride, as, on allowing to cool, more ammonia was absorbed. On treating the mass with dilute sulphuric acid, and rapidly

pouring off the solution obtained, a residue of less soluble substance was left, having all the external properties of nitride of iron prepared by other methods, and slowly soluble in sulphuric acid with formation of ferrous and ammonium sulphates. Nitride of iron is therefore formed by the action of ammonia upon ferrous chloride at the temperature of molten lead. It is impossible definitely to decide whether an intermediate compound, e.g., FeNH_2Cl , is formed at this temperature, as a mixture is obtained of nitride of iron, ammonium chloride, and unaltered ferrous chloride, which latter absorbs ammonia. The solution obtained by treating the contents of the tube with sulphuric acid, as described above, was tested for hydrazine by distilling with caustic soda, continuing as long as the contents of the distilling flask were liquid, and testing the distillate with Fehling's solution. A very slight reduction, obtained in one case, was found to be due to a trace of ferrous salt carried over.

In another case the sublimate alone was tested by precipitating with potash, filtering, and boiling with Fehling's solution. No reaction, however, was obtained.

When ferrous chloride is heated with ammonia, the latter is first absorbed to form the molecular compound mentioned above. On further heating, reduction of the ferrous chloride takes place, resulting in the formation of ammonium chloride, nitride of iron, and a small quantity of nitrogen. To prepare the nitride in quantity by this method, a temperature of about 600° appears to be necessary.

Ferrous bromide was prepared and similarly treated. The temperature of reaction was not sensibly lower, and the products of reaction were analogous. In one case, where ferrous bromide was used, the products of the reaction, at about 350° C., were dissolved in acid and reduced with zinc, previously to distillation with caustic soda, in order to prevent oxidation of any hydrazine, by means of the ferric hydrate formed. No trace of reduction of the Fehling's solution was obtained in the distillate from caustic soda.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART V.

By HARRY BREARLEY.

(Continued from p. 199).

SILICON.

THE items are arranged, as sharply as possible, under the following heads:—

- I. THE ESTIMATION OF SILICON IN METALS.
 - a. Iron and Steel.
 - b. Alloys.
 - c. Aluminium.
- II. THE ESTIMATION OF SILICA IN REFRACTORY MATERIALS.
- III. THE ESTIMATION OF SILICA IN SLAGS AND SILICATES.
- IV. MISCELLANEOUS.

I. THE ESTIMATION OF SILICON IN METALS.

I.a. In Iron and Steel.—

665. ABEL (C. N., vi., 124; and FORBES (C. N., xvi., 105).—Pig dissolved in HCl, evaporated; Si dissolved from the graphite with KHO, and recovered again by evaporation with HCl.
666. ALLEN (C. N., xxix., 91).—Similar to Abel. When iron is dissolved in HCl or H_2SO_4 , the silicon is not liberated as SiO_2 , but as leucon. For particulars of leucon see Wöhler (C. N., viii., 171 and 182).

567. SCHNEIDER (Y. C. S., lxi., ii., 162).—Dissolve in HCl, and evaporate with H_2SO_4 to dehydrate the silica.
568. BLUM (C. N., liii., 300).—Dissolve in HCl and evaporate. The addition of $(\text{NH}_4)\text{Cl}$ increases the rapidity of evaporation.
569. RUBRICUS (Y. I. S. I., 1893, i., 411).—Dissolve pig in small excess of HCl, filter at once, ignite residue with KNO_3 , boil with $(\text{NH}_4)\text{Cl}$, and collect the hydrated silica.
570. SCHNEIDER (Y. I. S. I., 1893, ii., 532).—Processes of evaporation with HCl, or solution of the baked residue in HCl, are erroneous. SiO_2 is markedly soluble in HCl: it should be dehydrated by evaporation with H_2SO_4 .
571. DROWN (C. N., xlii., 299).—On dissolving Fe in HCl, the less Si passes into solution the stronger the acid. High Si irons may be fused with KHSO_4 , digested with HCl, and SiO_2 filtered off.
572. ALLEN (C. N., xl., 65).—As leucon, the form in which Si is liberated from iron, is soluble in water, no fixed proportion of silicon invariably passes into solution. Means of distinguishing leucon given.
573. EGGEKZ (C. N., xvii., 100 and 115).—Dissolve Fe with Br or I and water at 0°C , and separate SiO_2 from the slag with $\text{Na}_2\text{CO}_3\text{Aq}$. If iron is dissolved in HCl, very little Si passes into solution; if HNO_3 , much. By dissolving in H_2SO_4 and evaporating to SO_2 fumes, nearly all the SiO_2 is separated.
574. EGGEKZ (C. N., xviii., 232).—Dissolve in H_2SO_4 ; add HNO_3 , evaporate, re-dissolve in HCl, and collect SiO_2 .
575. PIESSEZ (C. N., xxix., 57).—Effect solution with nitro-sulphuric and evaporate at 100°C .
576. DROWN (C. N., xl., 40).—Decompose with nitro-sulphuric and evaporate to SO_2 fumes. By treating iron with HCl, H_2SO_4 , and HNO_3 , the Si passing into solution is in the order named.
577. DROWN (C. N., lx., 20).—Nitric acid of sp. gr. 1.135 dissolves pig-iron completely without causing any SiO_2 to separate.
578. LANGMUIR (C. N., lxxix., 216).—To effect a more complete dehydration in (676) a strap is passed round the beaker, and the solution kept in motion over the naked flame.
579. JONES (C. N., lx., 79).—The nitro-sulphuric solution is evaporated in three to five minutes by two flames, one of which impinges on the surface of the liquid.
580. AUCHY (Y. I. S. I., 1898, ii., 559); and CROBAUGH (Y. I. S. I., 1899, ii., 484).—To use a mixture of aqua regia and H_2SO_4 is more accurate and convenient than nitrosulphuric acid.
581. JÖRTNER (Y. I. S. I., 1884, 603).—Comparison of various methods. Drown's process (676) gives good results in the presence of Ti. Estimating SiO_2 by volatilisation with HF is objectionable.
582. DUDLEY (C. N., lxx., 186).—Silica dehydrated by evaporation to SO_2 fumes is partially re-dissolved when allowed to stand in the acid solution.
583. FORD (Y. S. C. I., 1893, 1061).—Silica dehydrated by process 676 is not re-dissolved as Dudley suggests.
584. JERVIS (C. N., lxxviii., 63).—Dissolve in dilute nitro-sulphuric acid, and deliver water on to the dry hot mass; this avoids cooling cracks, and no SiO_2 is re-dissolved on standing.
585. TROILIUS (C. N., xlii., 310).—Dissolve in dilute H_2SO_4 , evaporate to SO_2 , dissolve separated salt in HCl, filter off SiO_2 , and wash with HNO_3 .
586. TURNER (C. N., lvi., 49).—Phosphoric irons evaporated with H_2SO_4 may give a white, but still impure, SiO_2 . When evaporated with aqua regia, the colour of the SiO_2 is an indication of purity.
587. TOSH (C. N., xvi., 168).—Evaporate aqua regia solution, fuse SiO_2 with KNO_3 , and evaporate the dissolved melt with HCl.
588. MORGAN (C. N., lvi., 221 and 244).—Evaporate aqua regia solution to syrupy condition only; the SiO_2 is free from phosphide of iron with highly phosphatic irons.
589. FARRY and MORGAN (C. N., lxxvii., 149).—As Morgan; but evaporate to dryness and then re-evaporate HCl solution to skin; fuse with KHSO_4 , to separate iron phosphide. H_2SO_4 is essential when SiO_2 is to be volatilised with HF.
590. LIEBRICH (Y. I. S. I., 1896, i., 532).—After evaporating acid solution of the pig, ignite the filtered residue with KHSO_4 to burn off graphite and separate Fe and Ti.
591. LECLERC (Y. C. S., lx., 1397).—Dissolve in nitro-hydrochloric acid, expel HNO_3 , and evaporate after addition of ammonium chloride to prevent Mn contaminating SiO_2 , and KCl to prevent the decomposition of Fe_2Cl_6 .
592. TURNER (Y. C. S., xlv., 260).—Various methods compared. Dissolving in HCl or nitro-sulphuric untrustworthy for Si pigs or Si spiegels.
593. MORGAN (C. N., lvi., 83).—Roast finely-divided sample in the muffle to oxidise Si, digest with HCl, and collect SiO_2 .
594. WATTS (C. N., xlv., 279); and TURNER (C. N., xlix., 233).—Volatilise Si along with iron in Cl at red heat, collect SiCl_4 in H_2O , and evaporate for SiO_2 . Any slag or cinder remains with the carbon in the boat. (See 673 and 748).
595. KONINCK (Y. I. S. I., 1894, i., 614).—The solution of the iron is precipitated by $(\text{NH}_4)\text{HO}$ or acetate, and the ignited precipitate heated in HCl gas; SiO_2 (and Al_2O_3) remain in the boat.

1.b. In Alloys.—

596. CLERC (Y. I. S. I., 1889, ii., 479).—Dissolve powdered ferro-silicon in bromised HCl, evaporate one-half, and collect SiO_2 ; the error is insignificant.
597. HOGG (C. N., lxxvii., 27).—Boiled finely-powdered Fe-Si or Si spiegel with nitro-hydrochloric acid, filter, and ignite to SiO_2 . Only 0.7 to 0.3 per cent Si passes into solution when dealing with 10 to 15 per cent alloys.
598. PLATT (Y. I. S. I., 1893, i., 411).—As Hogg, but adds H_2SO_4 containing SO_2 to render Si insoluble.
599. MURRAY and MAURY (Y. C. S., lxxii., ii., 599).—Decompose silico-spiegel with HCl + H_2SO_4 , evaporate to SO_2 fumes, and volatilise SiO_2 with HF. Done in half an hour.
700. WILLIAMS (Y. I. S. I., 1889, i., 397).—Fuse Fe-Si with Na_2CO_3 and evaporate melt with HCl; the SiO_2 is pure.
701. ZIEGLER (C. N., lxxvi., 295).—Fuse Fe-Si or Fe-Cr with KHSO_4 , dissolve melt, and collect SiO_2 .
702. DONATH and HALLSIG (Y. I. S. I., 1897, ii., 488).—Ferro-silicons cannot be dissolved by HCl, HNO_3 , or aqua regia. Fuse with Na_2CO_3 and NaNO_3 .
703. BORNTÄGER (Y. C. S., lxxvi., ii., 695).—Silicon is analysed by dissolving in potash; the impurities, Al_2O_3 , Fe_2O_3 , and Fe-Si, are left undissolved.
704. TATE (C. N., lxxx., 235).—Fuse Fe-Cr with Na_2O_2 , evaporate the neutralised melt, and heat with H_2SO_4 to fuming; finally, vaporise SiO_2 with HF. Much of the Cr escapes during the H_2SO_4 treatment as chlorochromic acid.
705. MOISSAN (Y. C. S., lxxvi., ii., 43).—Carbide of silicon (carborundum) is decomposed by fusion with KHO . KNO_3 and KClO_3 have no effect on it, nor has any mixture of acids.
705. MÜHLHAUSER (Y. S. C. I., 1894, 402).—Uses the elutriated powder of carborundum, and decomposes with KNaHO . Estimate the carbon by burning a like powder with PbCrO_4 .

* Gives a method of estimating graphite and combined carbon in ferro-silicons which should have been noticed under "Carbon."

707. SCHÜTZENBERGER (Z. C. S., lxii., 1050).—Carborundum as Moissan. The volatilisation of the Si in a current of chlorine is not complete.

I.e. In Aluminium.—

708. HUNT, CLAFF, and HANDY (C. N., lxx., 223).—Si exists in Al in the graphitoid and combined form. Dissolve in nitro-hydrochloric, evaporate with H_2SO_4 , and collect Si+ SiO_2 . SiO_2 cannot be estimated by volatilisation. Si is also partially oxidised, therefore fuse with Na_2CO_3 , dissolve in HCl, evaporate with H_2SO_4 , and weigh total Si as SiO_2 .

709. HANDY (Z. C. S., lxii., ii., 191).—To determine graphitic Si decompose Al with HCl+HF, filter through paraffin-coated funnel, fuse with Na_2CO_3 , and estimate as SiO_2 .

710. REGLSBROER (Z. C. S., lxiv., ii., 48).—Decompose with aqua regia + H_2SO_4 , drive off HNO_3 , collect Si+ SiO_2 , and evaporate with HF; loss = SiO_2 . Evaporate residue with HF+ HNO_3 ; loss of weight = Si. (See 708 and 761).

711. MOISSAN (Z. C. S., lxx., ii., 339).—Decompose with HCl, fuse insoluble and add to main solution; evaporate at $125^\circ C.$ to separate SiO_2 .

712. SIBBERS (Z. C. S., lxiv., ii., 409).—On dissolving Al in HCl appreciable amounts of Si are volatilised. Dissolve in current of H which on ignition at the delivery tube deposits SiO_2 .

713. GONTIÈRE (Z. C. S., l., 1896, 83).—Dissolve in nitro-hydrochloric and proceed as for steels, taking no account of graphitic Si.

714. ANON. (Z. I. S. I., 1892, i., 491).—Dissolve in KHO, acidulate with HCl, evaporate, and collect SiO_2 .

(To be continued).

ON THE ESTIMATION OF MANGANESE AS PYROPHOSPHATE.

By WILHELM BÖTTGER.

THE want of precise instructions on the use of Wolcott Gibbs's method (*Zeits. f. Analyt. Chem.*, [6], v., 174), the partial contradictions as to certain of its details (Fresenius, "Quant. Analyse," 259; Kessler, *Zeitsch. f. Anal. Chem.*, xxxiii., 8), and the want of success of the method when in unpractised hands, have decided the author to closely examine this method of quantitative estimation. Although the conditions necessary for the success of the operation have figured for some months past in Autenrieth's work on analysis, the results of his experiments have not yet been published.

In Miller and Kiliani's book on Chemical Analysis, as well as in that of Zimmermann, we read that a neutral solution of a salt of manganese should be treated with an excess of disodic phosphate, and the precipitate of $Mn_2(PO_4)_3$ obtained dissolved in a few drops of hydrochloric acid. The liquid should then be brought to boiling, an excess of ammonia added, and then heated on the water-bath until the double phosphate of ammonium and manganese, deposited in the amorphous state, becomes transformed into crystalline leaflets. Autenrieth's instructions differ from the above in the use of an ammoniacal salt and the absence of acid.

From the point of view of equilibrium in phenomena of supersaturation, the formation of a substance in the solid state takes place in any system when the concentration exceeds the solubility at any given temperature. If the dissolved material undergoes a modification (hydration, polymerisation, ionisation, &c.), the solubility, in the strictest sense, is in relation to the part which remains intact. The apparent solubility includes the modified portion. The concentration of the material is always connected by definite relations with that of the products of transformation. On bringing these two relationships together, that is, the one above mentioned, and

that which exists between the non-modified portion and the solid material, we can connect the definition of solubility with that of the concentration of the products of transformation. If we do this, particularly in the case of electrolytic decomposition into ions, we arrive at the conclusion that the product of the concentration of the ions under given conditions, and in presence of the solid phase, possesses a definite value.

The analytical expression (Ostwald, *Wissenschaftliche Grundzüge der Analytischen Chemie*, ii., aufl. 70; *Grundriss der Allgemeinen Chemie*, iii., aufl. 427) of this will be—

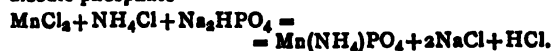
$$A^m \times B^n = \text{const.},$$

where A and B represent the concentration of the ions, and m and n the respective figures of the combining weights of the ions which take part in the transformation.

In the case in question where the manganese is precipitated in the state of $Mn(NH_4)PO_4$, it is necessary to bring about such conditions that the concentration of the ions PO^{4-} and NH_4 may be as great as possible.

The increase in the concentration of the ion NH_4 before the addition of the phosphate is effected by the use of an ammoniacal salt, which, like other normal salts, is capable of dissociation. As solutions of ammonia contain few NH_4 ions, it is not necessary to add a large quantity; besides that, $Mn(OH)_2$, which is then converted into phosphate, is easily formed. There is, however, a disadvantage in the fact that $Mn(OH)_2$ in absorbing oxygen passes into the state of $(MnOH)_3$, which completely destroys the success of the analysis.

The best means of preventing this annoyance consists in introducing into the neutral solution a quantity of ammoniacal salt* representing five to ten times the molecular quantity, boiling the liquid, and then adding an excess of disodic phosphate—



The free acid which is thus formed, and which prevents complete precipitation, is neutralised with a little ammonia. It is necessary to heat until the amorphous substance is completely transformed into a crystalline precipitate.

By examining the accompanying table, where the results of different analyses made with sulphate of ammonium and manganese are given, we can see that the method can be used perfectly well in all ordinary cases. One cause of error is due to the slight solubility of the precipitate in water, which can be shown by adding a little nitrate of silver to the filtrate.

No.	$Mn_2P_2O_7$ used. Grms.	Quantity found.	Per cent.	Observations.
1.	0.4449	0.4452	100.1	5 grms. of sulphate of ammonia, 25 c.c. of solution of disodic phosphate,* 10 c.c. of concentrated ammonia.
2.	0.4410	0.4401	99.8	
3.	0.4355	0.4371	100.3	
4.	0.4423	0.4417	99.9	
5.	0.4369	0.4383	100.3	
6.	0.4394	0.4412	100.4	Like 1 to 5, only 5 c.c. of dilute ammonia (double normal).
7.	0.4428	0.4432	100.1	
8.	0.4358	0.4351	99.8	3.2 grms. sulphate of ammonia, 25 c.c. Na_2HPO_4 solution, 10 c.c. dilute ammonia.
9.	0.4370	0.4363	99.8	
10.	0.4397	0.4388	99.7	Like 9, but 1.6 grms. of sulphate of ammonia.
11.	0.4362	0.4327	99.2	5 grms. sulphate of ammonia, with 10 c.c. of concentrated ammonia.
12.	0.4362	0.4327	99.2	10 cc. of dilute ammonia; precipitate washed with hot water.

* The solution of Na_2HPO_4 was about 12 per cent.

* The use of an ammoniacal salt has the advantage of increasing the concentration of the NH_4 ion, and, by preventing the dissociation of the ammonia, limiting the concentration of the HO ions.

Thus, as shown by analysis 11 and 12, the washing of the precipitate with warm water increases the error. It is therefore preferable to moisten the substance with slightly ammoniacal cold water.

If we take care to wash the phosphate until the filtrate leaves no residue when evaporated down and heated to redness on a strip of platinum, we can eliminate the ammoniacal salt by calcining over a blowpipe flame. The author has never found manganese in the residue from the evaporation of the wash-waters, on dissolving it with salt-petre and carbonate of soda. He concludes with the remark, that even when placed in inexperienced hands the method in question has been proved to be of advantage.—*Berichte*, xxxiii., p. 1019.

THE PLASTICITY OF SOLID BODIES, AND ITS CONNECTION WITH THE FORMATION OF ROCKS.

By W. SPRING.

FARADAY showed, in the year 1850, that two fragments of ice, pressed one against the other, united with greater ease as their temperature approached zero. This fact does not at first appear to be of any great importance, but soon—owing to the ingenuity of Tyndall—it soon led to the explanation of the formation of glaciers; his experiments are too well known to require further mention; suffice it to say that they established the fact that compression alone of the snow was enough to form those immense glaciers which cover many mountain tops and the polar regions.

It may be asked if this phenomenon is peculiar to ice, and whether other bodies do not also possess the property of joining together under pressure. The results of numerous observations show that such a property is to be found in different degrees in certain substances; it is very slight in some bodies, more pronounced in others, and is distinct in a few substances only.

The task of verifying the existence of this property appeared to be of great interest, inasmuch as important light would be thrown on several questions concerning the physical properties of solids in general, and more particularly on the formation of sedimentary rocks from the gravels, sands, and clays from which they are derived.

The manner in which the hardening of these rocks is effected is still obscure. Most of them appear to want in matter capable of acting as a cement uniting the agglutinated particles. How, then, can the coherence have been caused in so complete a manner in substances which were originally non-coherent? Might it not be by a process similar to that which formed glaciers? and, if so, would not our ancient rocks, with their folds, fractures, and faults, be found, from the point of view of their formation, in the image of our modern glaciers?

To make certain whether solids, other than ice, are endowed with the property of joining together, it suffices to bring about the conditions in which this regelation is produced. These conditions are pressure, temperature, and time. In other words, we must find out whether certain solid particles, unable to join together under ordinary conditions, cannot be made to agglutinate under the influence of a sufficiently great pressure.

We therefore constructed a compressor by which we could attain a pressure of about 10,000 atmospheres, or nearly 70 tons per square inch, in the cold, without any of the material escaping; also another apparatus, capable not only of exerting similar pressures, but also of being heated up to 400° C. at the same time.*

The first experiments showed that all bodies endowed with the property of changing their form under pressure without breaking became as solidly agglutinated as if they had been fused, while those which were not malleable remained pulverulent even under the greatest pressure.

It should be specially noticed that quartz or clayey sands, carbonate of lime in various forms, oxides of iron and alumina, in short the substances which most frequently enter the composition of rocks, are those in which agglutination was non-existent, or at any rate imperfect. The conditions for the solidification of rocks cannot therefore be found exclusively in pressure. It may be mentioned that, if a pressure of 70 tons is insufficient to agglutinate sand, the quartzose rocks must have been solidified by some other force than pressure alone, as 70 tons pressure represents a column of sand 50,000 metres high.

The metals behave in a striking manner; the more malleable they are, the better they agglutinate; they flow under the action of pressure, and grains (filings) are cemented into a solid mass as if they had been fused. It must not be supposed that they could have been fused, for the pressure was never applied by blows, or suddenly, but always very gradually. It was further observed, experimentally, that the rise of temperature was negligible. But if the hypothesis of fusion cannot be entertained to explain this cohesion, we can only attribute it to an inter-diffusion of the molecules in contact, to a sort of inter-solution of the surfaces of the solids. This explanation may appear to be a bold one; it is not difficult, however, to show that it agrees with observed facts.

If there is a true diffusion of the molecules at the surfaces of contact, it follows that the compression of different metals will make an alloy, and not simply a conglomeration of particles, each retaining its individual properties. Experiment has confirmed this supposition in the most complete manner.

By compressing a mixture of tin and copper, both in the form of powder, we obtained bronze; zinc and copper gave brass; copper and antimony furnished a characteristic violet alloy; finally, bismuth, tin, lead, and cadmium, in the proper proportions, formed an alloy which fused in boiling water.

It must be quite understood that, after the first compression of a mixture of the filings of metals, the alloy is only formed at the true surface of contact of the grains. To increase the quantity, the cylinder formed by compression is filed, and the filings compressed again; this is repeated five or six times, when the result is visible to the naked eye.

We must therefore admit that two fragments of solid bodies, of the same or even of different species, brought into absolute contact by the action of sufficient pressure, diffuse one into the other in the same way as a soluble body diffuses in its solvent. In a general way these experiments show that the phenomenon of solution is not subordinate to a state of liquidity of the material; solid bodies dissolve, giving solid solutions. J. H. van't Hoff has arrived at similar conclusions in examining the anomalies shown by the congealing of certain solutions. We must therefore regard solid bodies as having conserved, to a certain point, the molecular mobility characteristic of the fluid state. But even here there remains a notable difference, as liquids are subject to free evaporation, while solids appear to retain all their molecules in a definitely determined space appropriate to their special nature. For example, zinc will evaporate into copper, and *vice versa*.

If the agglutination of bodies is not only a mechanical act, but if it is rather the consequence of a reciprocal solution of solid bodies, it naturally follows that bodies not soluble in each other will not be joined together by pressure. This is proved to be so by experiment. We know that simply melted lead and zinc are not miscible; they separate one from the other like oil and water; it is

* The description of this apparatus will be found in the *Bulletin de l'Académie Royale de Belgique*, Series 2, vol. xlix., p. 523. Details of the experiments, of which a summary only is given in this article, will be found in the same publication, Series 3, vol. x., p. 204; vol. xxviii., p. 23; vol. vi., p. 307; vol. xiii., p. 409; vol. xxx., p. 199;

vol. xxxii., p. 51. See also the *Bulletin de la Société Chimique de Paris*, vol. xli., p. 485; vol. xlvii., p. 299, and the *Zeitschrift für Phys. Chemie*, vol. v., p. 322.

only at high temperatures that the reciprocal solubility of these two metals becomes apparent. Bismuth behaves to zinc in the same way as lead does; now, if we compress, in the cold, a mixture of powdered lead and zinc, or bismuth and zinc, we only obtain an agglomeration softer than before, but still not a homogeneous mass.

Nevertheless, it may be asked whether the junction of solids is not all the same the consequence of the inevitable kneading produced by pressure rather than of a solid solution. The solid grains might well behave like balls of clay which have been worked up and form a mass without diffusion taking any special part in the operation. This can be proved by experiment; it suffices to eliminate the compression, and to make certain whether the solids still join together when all precautions are taken to effect their perfect physical contact simply by superposition. For this purpose accurately made straight sections of cylinders of different metals, such as gold, platinum, silver, copper, zinc, lead, tin, bismuth, &c., were prepared. These cylinders were then placed one on the other in couples, with no other pressure than their own weight. A certain amount of adherence was at once noticed, even in the cold. The temperature was then raised, though it was always kept far below the melting-point (from 200° to 1600° below, according to the metal). The duration of contact varied from three to twelve hours, according to the metal. The result was most surprising; the metals of the same kind were joined together, so as to form but a single mass, and the joint could not even be detected after cleaning the surface in the lathe. The pairs of metals, which were of different kinds, were alloyed the deeper as their malleability was greater. Thus copper and zinc formed a layer of brass a quarter of a millimetre in thickness, while lead and tin were alloyed to a depth of nearly 6 millimetres. Finally, the metals not having the power of dissolving, viz., zinc and lead, zinc and bismuth, only exhibited the commencement of a junction, without having any strength of cohesion at all. But the resemblance between solid and liquid solutions does not stop here. If we mix a solution of potassic saltpetre and chloride of sodium, sodic saltpetre and chloride of potassium are formed, but only in limited quantities; a certain proportion of the reagents appear to become inactive or incapable of doing chemical work. The inverse of this experiment gives the same results. In both cases the stoppage occurs at the same chemical moment, namely, when the force which gives the impulse to one reaction is exactly balanced by that which causes the inverse reaction. This state of chemical equilibrium is also produced when we submit certain solid bodies of different chemical natures to compression. Thus, we first compressed a mixture of dry sulphate of barium and carbonate of sodium; then, as the inverse, a mixture of carbonate of barium and sulphate of sodium. The result was a double decomposition of both reagents, and this was limited in both cases. Thus it is proved that the chemical reactions which take place between solid bodies under great pressure obey the same law as the reactions which take place in solutions.

It is thus established that the reciprocal solubility of substances is not only a condition of their agglutination in the solid state, but also of their chemical combination under the influence of pressure. Without doubt this is the only condition necessary with regard to agglutination, but not in so far as it concerns chemical reaction.

(To be continued).

Society of Arts.—In consequence of the Annual Dinner of the Institution of Electrical Engineers being fixed for Monday, December 3rd, the second lecture of Professor Fleming's Cantor course on "Electric Oscillations and Electric Waves," announced for that date, will be postponed until the following day, Tuesday, December 4th, at 8 o'clock, to suit the convenience of members and others who might be prevented by the dinner from attending it.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING OCTOBER 31ST, 1900.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, November 9th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 216 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from O.A. 1st to O.A. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 216 samples examined by us during the month, one was returned as "turbid," all the others were clear, bright, and well filtered.

The rainfall at Oxford during October again shows a deficiency, though rain fell on fifteen days out of the month; the actual rainfall was 2.35 inches, the average fall is 2.75 inches; this leaves a deficiency of 0.40 inch for the month and brings the total deficit for the year up to 2.45 inches, or 11.5 per cent on the thirty years' average.

Our bacteriological examinations of 551 samples have given the results recorded in the following table; we have also examined 40 other samples, from special stand-pipes, &c., making a total of 591 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	184
New River, filtered (mean of 125 samples) ..	10
Thames, unfiltered (mean of 27 samples) ..	1441
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 301 samples) ..	26
Ditto ditto highest	336
Ditto ditto lowest	0
River Lea, unfiltered (mean of 27 samples) ..	212
River Lea, from the East London Company's clear-water wells (mean of 44 samples) ..	15

Of the 470 samples of filtered water supplied to London, examined bacteriologically during the past month, thirty samples, or 6 per cent of the whole number of samples examined, contained more than 100 microbes per c.c.

The fact that 6 per cent of the samples collected this month contained more than 100 microbes per c.c., as compared with 2 per cent last month, may be accounted for by the large increase of rainfall over that of the previous month; in September, as will be seen by our last report, only 0.41 inch of rain fell, while during October 2.35 inches have been recorded. At the same time we are able to state that a number of special daily examinations have failed to show the presence of any pathogenic microbes in any sample of filtered water.

It is understood that the moment we discover any sample from the clear-water wells which contains more than 100 microbes per c.c., or shows any trace of turbidity, we at once communicate with the Water Company, and inform

them that certain of their filters are working defectively, and ought to be instantly remedied.

We are, Sir,

Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 238).

Dilatometer Experiments (continued).

Experiment 24.—The same dilatometer was again heated slowly up to 217° , the following readings being obtained:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
117°	132	2 : 56	160°	225	3 : 24
130	161	3 : 02	172	250	3 : 39
140	182	3 : 14	182	271	3 : 49
150	203	3 : 18	201	311	4 : 15

The tube was then cooled down—

Temp.	Scale.	Time.
169°	248	4 : 27
155	219	4 : 32
120	145	4 : 52

then heated to 225° to effect fusion, after which the following readings were obtained on cooling down:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
225°	415	5 : 37	165°	290	5 : 57
185	336	5 : 41	150	257	6 : 01
—	332	5 : 42	130	212	6 : 12
—	332	5 : 50	69	82	6 : 18
180	322	5 : 52			

This shows that melted selenium in paraffin may be cooled down quite slowly without any transformation taking place, for the last of the above readings agrees with that found for the vitreous form in the preceding experiment where the bulb was cooled at once from 220° to 57° . In order that the paraffin might not solidify, the tube was placed over night in water which varied in temperature from 60° to 75° ; on the following morning, a reading was taken at 74° and the level was found to be 78, showing that the selenium had suffered contraction, but had not gone over entirely to the metallic form. On then raising the temperature, the following readings were made:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
74°	78	10 : 50	111°	127	10 : 30
85	100	11 : 02	111.5°	125	11 : 50
90	108	11 : 05	148	199	12 : 09
95	118	11 : 09	200	309	12 : 33
105	132	11 : 19	152	205	12 : 49
109	131	11 : 26			

These observations were made in diffused daylight. The experiment still further confirms the statement that metallic selenium undergoes no change in being heated to 200° , and that when the temperature is brought back to 150° the reading accords with that obtained with rising temperature.

The indications of a partial change at about 70° , followed by no further contraction up to 105° , are of interest, and a further experiment was undertaken later on in the hope of being able to repeat these observations. The following is the experiment in question:—

Experiment 71.—Selenium in solid paraffin. During three hours the temperature was slowly raised from 55° to 97° without any indication of a change; at this point contraction began; but the apparatus being left to itself, the temperature gradually fell during the succeeding four hours, to 73° ; the change had then proceeded to a con-

siderable extent; further heating at higher temperatures brought out the fact that below 90° the change, even when it has commenced, goes on very slowly—at 80° it amounted to about three scale divisions in an hour, where the total contraction involved nearly 80 scale divisions—but the drop was continuous and reached a limit after about fourteen hours' heating at temperatures between 80° and 90° . This experiment comes in a later class than the preceding, and was made with a weighed quantity of selenium and a dilatometer whose capillary arm had been previously calibrated. This method of procedure is much to be recommended for cases like the present, since it makes it possible to estimate at any moment approximately the density of the material studied.

The final density of the selenium was found in this way to be 4.83 . The extreme slowness with which the change goes on at 80° suggests that the observations of Experiment 24 may have no special significance at all; the change was probably incomplete and did not proceed rapidly to an end until the temperature was raised above 100° .

On the other hand, the existence of red crystalline forms of selenium, which, according to Muthmann, go over to metallic selenium at 110 – 120° , and which have a density of about 4.5 , points to the possibility of the occurrence here, exceptionally, of such a form; in that case Experiment 71 simply failed to reproduce the red form. It should be said, further, that no indication whatever of a change of vitreous selenium into the red crystalline modification was observed in any of the other dilatometer experiments, although such a change is known to go on superficially when vitreous selenium is brought into carbon disulphide at ordinary temperatures.

The following experiments were made with liquid paraffin unless otherwise stated:—

Experiment 26.—This determination was made as a further study of the density relations of the form of selenium which is produced when the melted element is cooled to 200° and maintained at that temperature until no further change of volume is observed. A dilatometer containing vitreous selenium was placed in a bath at about 150° ; at this temperature the change is immediate and gives rise, as is well known, to the ordinary metallic selenium. The experimental data are as follows:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
155°	260	12 : 12	219°	531	12 : 47
160	273	12 : 14	200	478	12 : 54
178	323	12 : 20	—	468	12 : 56
198	380	12 : 25	202	461	1 : 06
210	413	12 : 30	200	450	1 : 11
218	440	12 : 33	—	444	1 : 13
220	460	12 : 35	—	438	1 : 15
221	530	12 : 43			

At this point the temperature was allowed to drop for a few moments to 182° ; it was raised again at once:—

Temp.	Scale.	Time.
185°	370	1 : 25
198	386	1 : 31
201	393	1 : 32
196	380	2 : 06

The material was then heated again to fusion, and cooled to 200° :—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
201°	500	3 : 10	200°	443	3 : 49
199	490	3 : 11	200.5°	430	3 : 56
198	485	3 : 13	—	418	4 : 11
199	485	3 : 17	198	398	4 : 17
—	485	3 : 20	—	395	4 : 24
200	479	3 : 28	197	389	4 : 44
—	469	3 : 35	199	393	4 : 54
—	461	3 : 39			

This experiment shows clearly that the form produced at 200° is identical with the original metallic selenium. The slight difference in the readings at 200° before and after fusion, is negligible in comparison with the total

* From the *Journal of Physical Chemistry*, vol. iv.

change of level involved in the transformation. The heating at 220° to effect fusion was prolonged until there was little or no further change during several minutes, since it was plainly desirable to have the mass of selenium free from crystals of the metallic form; on the other hand, there was no object in refining too far upon this point, since we may say certainly that in the cooling down from 220° to 200° there would always be a small quantity of material deposited from the solution, either in the form of metallic crystals, or perhaps in some other modification if such were capable of existence at that temperature.

Experiment 28.—This experiment, which was made with the dilatometer used in Experiment 27, was undertaken to determine whether or not there are two substances in the melt when selenium is brought to complete fusion and then allowed to go over partially at 200°. If two forms are present in the liquid state, forming but one phase, then the melting-point will be lowered, and in consequence, at temperatures below—but very near—217°, any of the solid form present will melt and the dilatometer level will rise; if such is not the case it will continue to fall. The readings were as follows:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
80°	44	9:47	221°	558	12:01
100	92	9:54	200	497	12:06
190	350	10:32	201	469	12:39
207	398	10:50	199.5	447	12:48
218	433	11:02	214	483	12:54
220	530	11:29	215	478	1:08
—	553	11:53	214.5	474	1:15

It is plain from these figures that at 215°, under the conditions of the experiment, the contraction continued. The recorded temperatures are uncorrected; on comparing the thermometer used with a standard thermometer calibrated at the Reichsanstalt in Berlin, it was found that the one used in these experiments registered 3° too low at 215°. Since selenium expands on melting, it follows from the theorem of Le Chatelier that the melting-point will be raised by increased pressure; but as the prevailing pressure within the tube was, in this experiment, not above 6 to 8 atmospheres, it is improbable that this would very sensibly affect the melting-point. Reicher (*Recueil Trav. Pays-Bas*, 1884, ii., 262) found that an increase of 4 atmospheres only raised the inversion point for rhombic and monoclinic sulphur by about 0.2°, whereas the melting-points of various substances—water, naphthalene, &c.—are even less sensitive to pressure changes (Ostwald, "Lehrb." i., 1033). It seems most likely in this case that the recorded value 217° is somewhat low and that the melting-point of grey selenium should be placed nearer 220°. At any rate the experiment shows that liquid selenium continues to go over into the solid form at temperatures lying very near the melting-point, and this renders the existence of a second form in the liquid phase most improbable.

Experiment 29.—This determination was conducted with the same dilatometer that was used in Experiment 28. Starting from vitreous selenium, the following readings were made:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
55°	60	2:38	191°	418	2:49½
80	125	2:41	197	419	2:50
132	270	2:45	202	407	2:54
158	350	2:47	204	406	3:06
172	385	2:48	201	396	3:57
185	410	2:49	195	380	5:20

This experiment shows that it is sometimes possible, by rapidly raising the temperature of vitreous selenium, to heat it to about 185° before the transformation begins; but a comparison with the readings in Experiment 26 shows that the form produced in this way at 200° is the same as that obtained by any of the other methods used; and, furthermore, its density is not changed by heating for two and one-half hours at about 200°.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 21st, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

MESSRS. S. S. Napper, G. E. Tomlins, O. Silberrad, C. Watson, C. T. F. Watts, and H. F. F. B. Fermor were formally admitted Fellows of the Society.

The following certificates were read for the first time:—Messrs. George Lowe Bennett, 13, St. Domingo Vale, Egerton, Liverpool; Frederick Nisbett Binks, 2, Holywell Terrace, Millbourne Avenue, Drumcondra, Dublin; Herbert James Singleton Boyes, 9, Rua Episcopal, Sao Paulo, Brazil; Theodore Ridley Burnett, 83, Coltart Road, Liverpool; John Henry Cheesewright, 80, Sydney Road, Hornsey, N.; Albert Walker Comber, Rio Marina, I. d'Elba, Italy; Alexander Davidson, jun., 1, Almond Bank Terrace, North Murchiston, Edinburgh; Arthur Louis William Fechtner, 186, Spring Bank, Hull; Willie Ludford Freeman, 102, Marlboro' Road, Oxford; William Gasson, Kimberley, South Africa; George William Gibbings, Standard Bank of South Africa, Ltd., Salisbury, Mashonaland; John Gibson, Battle Hill, Hexham, Northumberland; Walter Augustus Handcock, Southbank, 40, Avenue Road, Highgate, London; William Arthur Hargreaves, Port Adelaide, South Australia; George Harker, 35, Boulevard, Petersham, Sydney, N.S.W.; Frank C. R. Hemingway, Albans, Forest Road, Walthamstow; John Brownlie Henderson, Brisbane, Queensland; Samuel Hewitt, 3, Chester Street, Norwich; William Henry Hewitt, 115, Fentiman Road London, S.W.; Adolf Jappé, Broad Oak, Oak Avenue, Bradford; Humphrey Owen Jones, Clare College, Cambridge; George Washington Kilner, 14, St. John's Park, Upper Holloway, N.; Morris Charles Lamb, Herold's Institute, Drummond Road, Bermondsey, London, S.E.; George Druce Lander, 1, Balmoral Road Nottingham; William McCall, Rio Marina, Isola d'Elba, Italy; Thomas Marginson Nightingale, 375, Bridgman Street, Bolton; Alexander Parry, Pietermaritzburg, Natal, S. Africa; John Paul, Bacteriological Institute, Grahamstown, Cape Colony; Ernest Vivian Pearce, The Beaches, Hayle, Cornwall; Henry Ernest Stapleton, St. John's College Oxford; Arthur Lambert Thornton, 2, Park Street, Bolton; Ferdinand Gerhard Wiechmann, 771, West End Avenue, New York; George Sampson Valentine Wills, Southwood, Croyham Road, South Croydon; Walter Bourne Woodbridge, Grey Friars, Chichester; Herbert Edwards Wright, 1, Brewers Street, St. Aldates, Oxford.

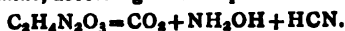
Of the following papers those marked * were read.

* 132. "*Action of Alkalis on Nitro-compounds of the Paraffin Series.* Part II. *The Reactions and Constitutions of Methasonic Acid and the Formation of Isoxazoles.*" By WYNDHAM R. DUNSTAN, F.R.S., and ERNEST GOULDING, B.Sc.

In a previous paper (Dunstan and Dymond, *Trans.*, 1891, lix., 410) it was shown that trimethyl isoxazole results from the action of alkalis on nitroethane, and triethylisoxazole from a similar action on nitropropane. Nitromethane, however, furnished no isoxazole, neither did secondary nitropropane.

Further investigation has shown that the action of alkalis, preferably ammonia, on nitromethane leads to the production of the substance briefly described by Lecco under the name of *methasonic acid*, $C_2H_4N_2O_3$, and regarded by him as an anhydride of nitromethane. The authors have obtained this substance in colourless crystalline plates melting between 60° and 70°, and have prepared and described several of its salts. Both the acid and its salts, $AgC_2H_3N_2O_3$, $NH_4C_2H_3N_2O_3$, &c., are unstable, and are liable to explode when suddenly heated.

When heated with acids or alkalis, methazonic acid breaks up into carbon dioxide, hydrogen cyanide, and hydroxylamine, according to the equation—

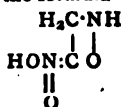


It is proved that 1 atom of nitrogen appears as hydrogen cyanide, and the other as hydroxylamine, whilst the carbon is equally divided between the carbon dioxide and the hydrogen cyanide.

On oxidation with permanganate, chromic acid, or hydrogen peroxide, methazonic acid furnishes carbon dioxide, hydrogen cyanide, and nitric acid. On reduction, either in acid or alkaline solution, with weak or strong reducing agents, the principal products are ammonia and formic acid. No trace of ethylene-diamine or methylamine could be found.

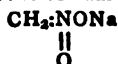
Alkyl derivatives of methazonic acid can be prepared, but they are highly unstable.

The authors propose the formula—



for methazonic acid as satisfactorily accounting for its reactions.

Adopting the view of Nef that the "salts" of the nitro-paraffins are to be regarded as derived from a tautomeride, and writing the formula of sodium nitromethane as



the authors show how methazonic acid is formed by a process of intermolecular oxidation followed by condensation of the nitromethane residue with formaldoxime simultaneously produced,—



By a similar process of intermolecular oxidation, followed by condensation with an oxime, it is shown how trimethylisoxazole, acetonitrile, and sodium nitrite are formed by the action of alkalis on nitroethane and how triethylisoxazole and similar products result from the action of alkalis on primary nitropropane; also, how secondary nitropropane gives no isoxazole, but acetone, nitrite, and hydroxylamine.

In this discussion it is assumed that nitro-compounds may be reduced to oximes (Dunstan and Dymond, *Proc.*, 1894, 139), and grounds are now stated for concluding that whilst the nitroparaffins themselves may be reduced (in acid solution) to substituted hydroxylamines, their salts, when reduced (in alkaline solution), furnish oximes or their decomposition products. These facts afford further justification for the view that the "salts" of the nitro-paraffins are derivatives of a tautomeride containing the group—



*133. "*Hexachlorides of Benzonitrile, Benzamide, and Benzoic Acid.*" By FRANCIS EDWARD MATTHEWS.

Benzonitrile mixed with water is saturated with chlorine and exposed to light till the yellow colour of the chlorine disappears. This process is repeated four or five times. The mixture is then subjected to steam distillation till all the benzonitrile has passed over. A thick oil remains, which, on purification by solution in and subsequent recrystallisation from, glacial acetic acid, gives colourless crystals of benzonitrile hexachloride, $\text{C}_6\text{H}_5\text{C}_6\text{Cl}_6\text{CN}$, melting at 157° .

The hexachloride is not easily hydrolysed, but heating with strong sulphuric acid at 170 – 180° converts it into the hexachloride of benzamide,

This, crystallised from acetic acid, melts at 187 – 188° . On treating the hexachloride of benzamide with fuming nitric acid, the following change takes place:—



The hexachloride of benzoic acid has been obtained pure, and has been analysed.

Its most striking property is the decomposition it undergoes on boiling with water, thus:—



The benzoic acid hexachloride and the monochlorobenzene tetrachloride are being further investigated.

*134. "*The Influence of Solvents on the Rotation of Optically Active Compounds.*" I. By T. S. PATTERSON.

Experiments to determine the influence of some solvents on the rotation of ethyl tartrate at various concentrations and temperatures were described. From the data obtained it is possible to deduce the rotation in each of the solvents dealt with, at any temperature within the limits of the experiments and at any concentration whatever, with fair accuracy.

The results obtained may be summarised thus:—

1. *Water.*—Ethyl tartrate in dilute aqueous solution has a much higher specific rotation than in the pure state. In solutions weaker than 55 per cent the rotation diminishes with increase of temperature.

2. *Methyl Alcohol.*—In dilute solution in methyl alcohol the specific rotation of ethyl tartrate is considerably higher than in the pure state.

In all solutions the specific rotation increases with increase of temperature, and at much the same rate as that of the pure ester.

3. *Ethyl Alcohol.*—Dilute solutions of ethyl tartrate in ethyl alcohol have a specific rotation slightly higher than that of the pure ester.

A maximum value of the specific rotation only seems to be reached at infinite dilution.

Increase of temperature causes increase of rotation in all solutions, the rate of variation being much the same as in the pure ester.

4. *Propyl Alcohol.*—No mixture of ethyl tartrate and propyl alcohol seems to have a higher specific rotation than pure ethyl tartrate at temperatures below 30° . At higher temperatures this is not the case.

The specific rotation in all solutions increases with increase of temperature, the rate of increase being generally slightly greater than in the pure ester.

5. *Glycerine.*—Dilute solutions of ethyl tartrate in glycerine have a considerable higher specific rotation at low temperatures than the pure ester, but as in the case of ethyl alcohol, the maximum rotation appears to be reached only at infinite dilution.

The influence of increase of temperature is in all cases to increase the rotation, the rate of increase being in dilute solutions less and in concentrated solutions greater than for the pure ester.

The data obtained were then discussed in order to trace, if possible, the variation in rotation of the dissolved ethyl tartrate to some known property of the solvent. It was suggested that the property known as internal pressure possesses the necessary qualifications, and investigation seem to justify its choice. Since the data obtained render the calculation of molecular solution volume possible, this was first compared with the rotation, and it was shown that a regularity can be established, and that the variation of rotation with change of temperature may be explained by certain assumptions regarding the variation of the asymmetry of a molecule with the variation of its volume.

These phenomena of rotation were then traced back a step further to internal pressure, or what is probably the same thing—heat of disaggregation, when very similar regularities are observed, which seems to show that the original assumptions, regarding the dependence of solution volume on internal pressure and of rotation on both, are justified.

*135. "The Action of Heat on Ethyl Sulphuric Acid."
By WILLIAM RAMSAY and G. RUDORF.

Ethyl hydrogen sulphate, when heated, yields as gaseous products of decomposition, sulphur dioxide, carbon dioxide and monoxide, and ethylene. The oxides of carbon, after the reaction has fairly started, are present in approximately equivalent proportions; this seems to point to the oxidation of the alcohol to oxalic acid, which is at once decomposed by the sulphuric acid. But glycol, heated with sulphuric acid, yields no carbon monoxide, hence this explanation is of questionable validity. When ethylene is bubbled through hot sulphuric acid, the products are the same in kind, and approximately the same in relative amount, as when hydrogen ethyl sulphate is heated. It was proved, in conclusion, that even at 250° carbon monoxide does not deprive sulphuric acid of oxygen; hence the formation of carbon dioxide in the preceding experiments cannot be attributed to the oxidation of carbon monoxide; it must have been a direct product of the reaction.

*136 "Contributions to the Knowledge of Fluorescent Substances. I. The Nitro-derivatives of Fluorescein."
By J. T. HEWITT and B. W. PERKINS.

Attention has been called to the non-fluorescence of the alkaline salts of tetranitrofluorescein (R. Meyer, *Zeit. Phys. Chem.*, 1897, xxiv., 468; and Hewitt, *Proc.*, 1900, xvi., 3; and *Zeit. Phys. Chem.*, 1900, xxiv., 1). This absence of fluorescence might possibly be explained by the existence of such tautomerism between the hydroxyl and nitro-groups as usually occurs with ortho- and para-nitrophenols, which would inhibit the double symmetrical tautomerism which, as one of the authors has previously pointed out, is so characteristic of many fluorescent substances. Under these circumstances it seemed very desirable to subject dinitrofluorescein to a further study, since this substance has only been analysed as a hydrate, $C_{20}H_{12}(NO_2)_2O_6$ (von Baeyer, *Annalen*, 1876, clxxxiii., 32). In order to obtain the true anhydrous dinitrofluorescein, its diacetyl derivative, which may be prepared by the action of acetic anhydride on the hydrate, was hydrolysed with fairly concentrated sulphuric acid (80 per cent). The anhydrous compound thus obtained dissolves in cold dilute soda solution with an orange-brown colour and with no trace of fluorescence. On warming, the solution readily goes blue, a salt of the hydrate being produced.

To determine the position of the nitro-groups in dinitrofluorescein, potash fusion was resorted to, and a small quantity of a substance of m. p. 114° (uncorr.) obtained. Nitroresorcinol (OH : OH : NO₂ = 1 : 3 : 4) melts at 115°.

Tetranitrofluorescein has also been examined, but the authors have been unable to obtain von Baeyer's numbers on analysis, the results always pointing to the formula $C_{20}H_{10}(NO_2)_4O_6$. Hence arguments deduced from the non-fluorescence of alkaline salts of this compound have no bearing on the question as to whether a pyrone ring is a "fluorophor" as the pyrone ring does not exist in the compound. Hence the results obtained with regard to the non-fluorescence of the alkaline solutions of dinitrofluorescein are especially significant, since in this compound the pyrone ring may be preserved intact and the fluorescence inhibited by the nitro-groups alone.

*137. "Derivatives of Ethyl α -Methyl- β -phenylcyanoglutarate."
By W. CARTER and W. TREVOR LAWRENCE.

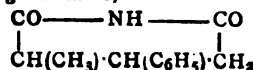
When ethyl cinnamate and ethyl sodiocyanacetate are allowed to interact in alcoholic solution, methyl iodide being subsequently added to the condensation product, it is found that the neutral reaction mixture consists almost entirely of equal quantities of the two stereoisomeric forms of ethyl α -methyl- β -phenyl- α -cyanoglutarate, $C(CO_2Et)(CH_3)(CN) \cdot CH(C_6H_5) \cdot CH_2(CO_2Et)$.

The α -modification is a crystalline solid, crystallising from ligroin in prisms which melt at 89°; the β -modification is a liquid boiling at 260° (100 m.m.); both isomerides, on addition of alcoholic potash, are converted into micro-

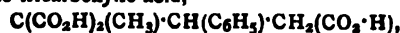
crystalline potassium salts, which separate completely from the mother-liquor.

From these potassium salts two distinct acids are obtained. The α -acid is fairly soluble in water, from which it separates slowly in prisms, melting with decomposition at 161°; the β -acid is much less soluble in water and melts at 194°.

Both acids are converted by hydrochloric acid into methyl-phenyl-glutarimide,—



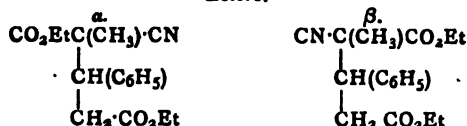
(prisms from water, m. p. 144°), and are completely hydrolysed by sulphuric acid with formation of methyl-phenyl-glutaric acid; on prolonged boiling with aqueous potash, they are converted into α -methyl- β -phenyl- α - γ -propane-tricarboxylic acid,—



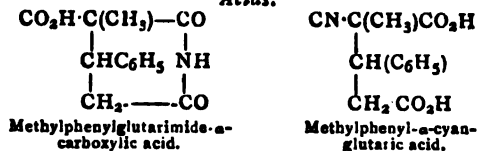
which crystallises from a mixture of chloroform and acetone, and melts, with decomposition, at 148°.

Acetyl chloride offers a means of differentiating between the two modifications, as it converts the α -acid into a substance melting at 110°, which possesses acid properties, and is easily decomposed by water, whereas the β acid forms a neutral substance melting at 146°, which is fairly stable towards water. The following formulæ have been assigned to the α - and β -esters, acids, and acetyl derivatives:—

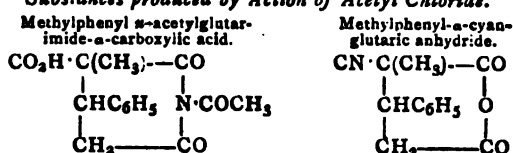
Esters.



Acids.

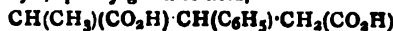


Substances produced by Action of Acetyl Chloride.



By this reaction methylphenylglutaric acid, previously obtained with great difficulty (Avery and Fossler, *Amer. Chem. Journ.*, 1899, xx., 516), may be easily prepared.

α -Methyl- β -phenylglutaric acid,—



crystallises from water or ligroin in prisms (m. p. 125°); theoretically, the acid should exist in two modifications, but up to the present time the authors have only isolated one form, which from its easy conversion into the anhydride, is probably the *cis*-modification.

Acetyl chloride converts the acid into a double anhydride with acetic acid, which crystallises from benzene in asbestos-like masses, melting at 107°; its formula is $[C(CO \cdot O \cdot COCH_3)H(CH_3) \cdot CH(C_6H_5) \cdot CH_2 \cdot CO]_2O$. When this is distilled, the true anhydride of methylphenylglutaric acid is obtained as a gum which slowly solidifies to a solid mass melting at 74°; the corresponding anilic acid is also a gum.

Methylphenylglutaric acid is unacted upon by boiling permanganate solution; boiling with fuming nitric acid converts it into two nitro-derivatives, of which the less soluble melts at 208° and the more soluble at 179°; both

substances possess the formula $C_{12}H_{13}O_4NO_2$, and are probably *o*- and *p*-nitrophenylmethylglutaric acid.

138. "The Nitration of Acetamino-ortho-phenyl Acetate (Diacylorthoaminophenol)." A correction. By RAPHAEL MELDOLA, F.R.S., and ELKAN WACHLER.

The object of this note is to correct and extend the statement (Meldola, Woolcott, and Wray, *Trans.*, 1896, lxi., 1325) that the nitration of acetamino-ortho-phenyl acetate under the conditions specified gave a mononitro-derivative, which, on further nitration, gave a dinitro-derivative. A repetition of this work has led to the conclusion that a dinitro-derivative is produced under all conditions of nitration. The statement in the paper above referred to was based on an error of analysis. The dinitro-derivative crystallises from hot water in long, ochreous needles melting at 201° , and softening at a temperature some degrees lower.

Two experiments gave $N=17.30$ and 17.39 per cent respectively. $C_6H_5(NO_2)_2NHAc \cdot OH$ requires $N=17.42$ per cent.

One acetyl group is split off during nitration. The dinitro-derivative is readily hydrolysed by boiling for a short time with sodium hydroxide solution, and the dinitroaminophenol thus obtained proved to be *picramic acid* as shown by the melting point ($167-168^\circ$), by conversion, by the diazo-method, into 2-chloro-4:6-dinitrophenol of m. p. $110-111^\circ$, and by a comparison of the properties of the diazoxide with the description of this compound given by Griess (*Annalen*, cxiii. 205).

The nitration of diacyl orthoaminophenol thus gives rise to the direct formation of 2-acetamine-4:6-dinitrophenol, and the formulæ given in that section of the the previous paper must be revised accordingly.

(To be continued).

MANCHESTER LITERARY AND PHILOSOPHICAL SOCIETY.

October 16th, 1900.

Prof. HORACE LAMB, F.R.S., President, in the Chair.

PROF. H. B. DIXON, F.R.S., communicated a summary of the results of experiments, conducted in conjunction with Mr. F. W. RIXON, B.Sc., on the "Specific Heat of Gases at High Temperatures."

As part of a larger investigation, the authors have determined directly the specific heat of carbonic acid, up to 400° C., at constant volume. The gas is screwed up in a mild steel cylinder, which is heated in a gas oven running on rails. The oven and cylinder can thus be brought quickly over the calorimeter, into which the cylinder falls through trap doors forming the bottom of the oven. The transference is thus effected with a minimum loss of heat. The difficulties arising from splashing and from escape of steam are overcome by dropping the cylinder into a glass tube dipping some distance below the water. The glass tube breaks at a crack made in the neck, and thus ensures a complete immersion of the hot cylinder at a good depth in the water, which closes over the cylinder in a cataraet.

A similar experiment being performed with the empty cylinder, the difference gives the heating effect of the gas.

The results given below for CO_2 show that the method, which it is hoped may still be improved, is a workable one.

Initial temp. of gas.	Final temp.	Mean.	Spec. heat.
115	16	65.5	0.200
192	16	104	0.211
298	21	159.5	0.288
398	21	209.5	0.356

The authors are now measuring the specific heat of nitrogen in the same way.

NOTICES OF BOOKS.

Flesh Foods, with Methods for their Chemical, Microscopical, and Bacteriological Examination. By C. AINSWORTH MITCHELL, B.A. (Oxon.), F.I.C., F.C.S. With Illustrations and a Coloured Plate. London: Charles Griffin and Co., Ltd. 1900. Pp. 336.

THE author has herein collected and summarised in a convenient form, records of investigations which are for the most part scattered throughout English and foreign scientific books and periodicals, and has selected those methods which appeared to be most suitable for the examination of meat and its preparations. The book forms an excellent practical handbook for medical men, analysts, inspectors, and others who may be called upon to judge the quality of various samples of flesh foods and derivatives.

The value of flesh as a food, apart from the nature of the animal it is taken from, is largely based on its colour, consistency, &c., on the proportion of fat, and on its taste and smell. It is not, however, possible to detect diseased meat with any certainty by chemical means, and it requires a good deal of practice to form a correct judgment from its appearance; still, there are certain tests, and they are fully described here.

The manufacture of meat extracts is a branch of trade which has increased enormously of late years; it is, however a great, though common mistake, to think that these widely advertised products are of much, if any value as food; Liebig expressly stated that his extract of meat was to be regarded as a stimulant, like tea or coffee, and not as a food, and his view is in the main confirmed by the experiments of later chemists; C. Voit, for instance, asserts that extract of meat is practically useless as food. In some products, eight or ten per cent of meat fibre is added with a view to giving them some food value, but it is obvious that a large quantity would have to be absorbed to get even as much nourishment as there is in an egg.

By means of bacterial examination it is often possible to supplement the information to be obtained from the chemical and microscopical examination of flesh. All the various forms of bacteria, known as *bacilli*, *micrococci*, and *spirilla*, may be found in sound or diseased meat, and in the chapter on this subject the author fully describes the more important ones. Sausages appear to be particularly deadly, some fresh uncooked ones have been found to contain over 6,000,000 microbes per grm. of meat, and as many as 9000 per grm. after being boiled for thirty minutes.

On the other hand, it is cheering to read that, according to Herdmann and Boyce, the typhoid bacillus does not increase in the tissues of the oyster, but perishes in its intestines; sea water also appears to be inimical to its development. Taking shell-fish in general, the *Bacillus coli communis* was sometimes found by these observers in those sold in towns, but there was no evidence as to its occurrence in molluscs taken from pure water. In no instances were any organisms giving all the reactions of *B. typhosus* isolated, though some of the bacilli resembled them in certain characteristics.

The book is well written and contains a fund of valuable and interesting information, and is provided with a very complete index of forty-eight columns.

Industrial Chemistry in Germany. ("L'industrie Chimique en Allemagne"). Its Economic, Scientific, and Commercial Organisation. By A. TRILLAT. In One Volume, Illustrated. Paris: J. B. Baillière and Sons. 1900. Pp. 500.

THE principal points dealt with by the author are the commercial prosperity of Germany, which he attributes to technical education and commercial organisation, the action of the German Chambers of Commerce, their patent laws, &c.

The first part of the book deals with the general situation in Germany; in the second part, which is of the greatest interest, the author describes the great chemical industries, such as metallurgy, soda processes, the manufacture of acids and alkalis, chemical products and drugs, organic and mineral colouring matters and allied subjects, artificial manures, ammoniacal salts, explosives, sugar making, porcelain and glass-working, electro-chemical and electro-metallurgical processes, &c.

The third, fourth, and fifth parts take in detail the economic and scientific organisation of chemical industry, and include an examination of the causes which have contributed to the progress of these arts in Germany.

A perusal of this book will be of interest both to practical men and to those who have to do with the organisation of laboratories and teaching institutions, and many useful hints will be gained therefrom.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 18, October 29, 1900.

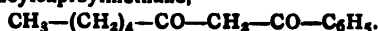
Ammoniacal Arsenates of Nickel.—O. DUCRU.—The author's researches establish the existence of four arsenates of nickel:— $(\text{AsO}_4)_2\text{Ni}_3 + 1.5\text{H}_2\text{O}$, $(\text{AsO}_4)_2\text{Ni}_3 + \frac{1}{2}\text{NH}_3 + \text{H}_2\text{O}$, $(\text{AsO}_4)_2\text{Ni}_3 + \frac{1}{2}\text{NH}_3 + \frac{1}{2}\text{H}_2\text{O}$, and $(\text{AsO}_4)_2\text{Ni}_3 + \frac{1}{2}\text{NH}_3 + \frac{1}{2}\text{H}_2\text{O}$. These latter three ammoniacal arsenates of nickel correspond to those of cobalt, and are derived from annabergite by the replacing of H_2O by NH_3 molecule by molecule. To prepare these ammoniacal arsenates of nickel and cobalt, it is necessary to use solutions in which the amount of free ammonia has been determined. The reaction takes place in sealed tubes. Under these conditions only small quantities of the products are obtained. The author therefore found it advisable to use specially constructed hermetically sealed flasks, in which 400 to 500 c.c. can be heated at once. The whole was placed on an air-bath at 135°C ., and surrounded by metal-foil.

The Selenides of Cobalt.—M. FONZES-DIACON.—By the action of selenium vapours on red-hot cobalt, Little obtained the protoselenide, CoSe , in an amorphous condition. The author prepares a series of cobalt selenides analogous to those of nickel, according to the conditions under which the reaction takes place. These are respectively CoSe_2 , Co_2Se_3 , Co_3Se_4 , CoSe . At a high temperature, hydrogen transforms these substances into a sub-selenide, Co_2Se , which, when left to itself, gradually loses its selenium. Cobalt selenate, when reduced by hydrogen, gives a series of oxy-selenides or mixtures of selenide and metallic cobalt, according to the temperature at which the reduction takes place.

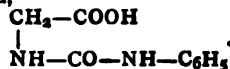
Modification of the Chemical Properties of some Simple Bodies by the Addition of very small Quantities of Foreign Materials.—Gustave Le Bon.—During researches on the various forms of phosphorescence, the author discovered that when minute quantities of foreign matter were added to certain substances, a combination was formed which profoundly modified the physical properties of these bodies. These changes in the physical state of various substances led to the question as to whether the chemical properties of certain bodies were equally modified by the presence of traces of foreign matter. The author's researches were made upon mercury, magnesium, and aluminium. In all cases a decided modification was observed when minute traces of mercury were added to magnesium or aluminium.

Cellulose, Mercerised Cellulose, Precipitated Cellulose, Hydrocellulose.—Léo VIGNON.—Cellulose and some of its modifications form the base of natural vegetable textiles and of artificial textiles whose substance is of vegetable origin. The author examined comparatively, from a chemical point of view, cotton cellulose, mercerised cellulose, cellulose dissolved in Schweitzer's reagent, and precipitated by acids, and Girard's hydrocellulose, with regard to their reducing properties, velocity of saccharification, and heats of combustion. He concludes from the numbers obtained that concentrated alkalis, such as are used in the operation of mercerising and probably hydrating, depolymerise the cellulose, without conferring on it new chemical functions. The same thing is true of dilute acids acting under the conditions necessary for the formation of Girard's hydrocellulose.

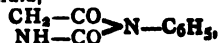
Two Acetones of the Acetylenic Series. Acetylonanthylidene and Benzoylonanthylidene. Transformation into β -dicetones by Hydration.—Ch. MOUREU and R. DELANGE.—The action of acetyl chloride and benzoyl chloride on sodium onanthylidene gives two acetylenic acetones. Acetylonanthylidene, $\text{CH}_3-(\text{CH}_2)_4-\text{C}\equiv\text{C}-\text{CO}-\text{CH}_3$, and benzoylonanthylidene, $\text{CH}_3-(\text{CH}_2)_4-\text{C}\equiv\text{C}-\text{CO}-\text{C}_6\text{H}_5$. These two new compounds, under the influence of sulphuric acid, fix a molecule of water, giving two new β -dicetones: acetylcaproylmethane, $\text{CH}_3-(\text{CH}_2)_4-\text{CO}-\text{CH}_2-\text{CO}-\text{CH}_3$, and benzoylcaproylmethane,—



Transformation of α -Amide Acids into Phenylhydantoins.—A. MOUNEYRAT.— α -Amide acids combine in alkaline solution with phenyl isocyanate, giving acid phenylureides; with glycol, for example, acetic phenyluride is obtained,—



Phenylhydantoin, prepared by means of glycol, corresponds to the formula,—



and is identical with the substance which Guareschi prepared by combining glycine with phenylurea. The author obtains analogous substances with alanine, α -aminobutyric acid, leucine, and phenylalanine.

Gaseous Exchange between the Atmosphere and Entire Plants.—Th. SCHLOSSING, junr.—The author's experiments were conducted on plants when their only source of nitrogen was from ammoniacal salts. He finds that plants are capable of using ammoniacal nitrogen in almost the same way as nitrogen of nitric origin. As in all preceding experiments, he finds that entire plants evolve more oxygen than that due to the carbon dioxide they decompose; this being due to the reduction of mineral salts drawn from the soil.

Researches on the Cystine in Contaminated Waters.—M. MOLINIÉ.—The author's researches lead him to the conclusion that all waters having an acid reaction, even distilled water, when submitted to Causse's reagent, give an orange colouration, not destroyed by sulphuric acid.

MISCELLANEOUS.

Messrs. Perken, Son, and Co., Limited, have just issued a new Catalogue of photographic apparatus, magic lanterns, and accessories, among which may be specially mentioned their "Optimus" lenses of various kinds, such as "europsopes," wide angle and portrait lenses, cinematographs, &c. Attention is also drawn to the "Ubique" hand camera, and the "Optimus" portable sets of photographic apparatus, which may be depended on for giving good results.

Action of Sulphurous Anhydride on the Metallic Sulphates, and especially on Ferric Sulphate.—U. Antony and E. Manasse.—In the same way as sulphurous acid reacts on sulphate of ruthenium giving a dithionate, so does it act on ferric sulphate. This salt in a 2 per cent solution, that is to say, at the limit of concentration where hydrolysis commences, absorbs SO_2 in the cold. The excess of gas is driven off by a current of CO_2 , and the whole of the SO_4H_2 precipitated from the solution by hydrate of barium, and the excess of barium by exactly the right quantity of CO_2 . The filtered solution contains dithionate of barium; if we treat it with KMnO_4 until the colour remains when hot, then with HCl to decolorise it, we get a precipitate of sulphate of barium, and on filtering it is easily seen that the solution still contains free H_2SO_4 ; this proves the presence of the dithionate in the liquid. The concentration of the ferric solutions, and pressure, have very little influence on the return; on the other hand, temperature has a considerable influence; at 0° we obtain about 80 per cent, falling to nothing at all at 95° .—*Gazz. Chim. Ital.* (1), xxxix., p. 483.

MEETINGS FOR THE WEEK.

MONDAY, 26th.—Society of Arts, 8. (Cantor Lectures). "Electric Oscillations and Electric Waves," by Prof. John A. Fleming, M.A., D.Sc., F.R.S.
WEDNESDAY, 28th.—Society of Arts, 8. "Malaria and Mosquitoes," by Major Ronald Ross.

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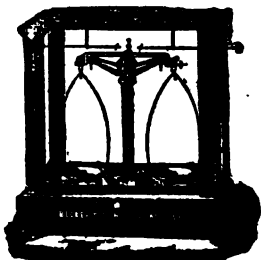
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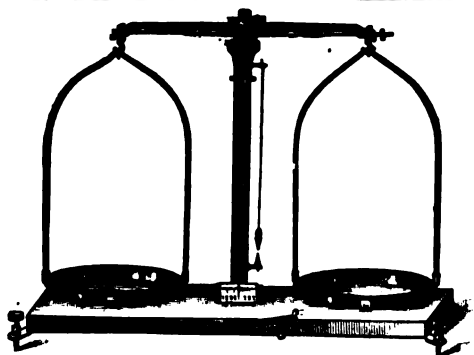
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2140.

ARGON AND ITS COMPANIONS.*

By WILLIAM RAMSAY, F.R.S.,
and
MORRIS W. TRAVERS, D.Sc.

THE discovery of krypton and neon was announced to the Royal Society in the early summer of 1898; and, subsequently, atmospheric air was found to contain a heavier gas to which the name of xenon was applied. Mr. Baly, in the autumn of the same year, called attention to the presence of helium lines in the spectrum of neon, an observation which confirms that made by Professor Kayser, of Bonn, and by Dr. Friedländer, of Berlin.

At the same time we imagined that we had obtained a gas with a spectrum differing from that of argon, and yet of approximately the same density: to this gas we gave the name metargon. It has now been found that the presence of the so-called metargon is to be accounted for by the fact that in removing oxygen from the mixture of these gases, which was then in our hands, phosphorus containing carbon was employed; this mixture when burned in oxygen yields a spectrum to some extent identical with that furnished by carbon monoxide, but differing from it inasmuch as lines of cyanogen are also present. We have no doubt that the so-called metargon, the spectrum of which is visible only at high pressure, and only when impure phosphorus has been employed to remove oxygen, must be attributed to some carbon compound. In spite of numerous experiments we have not yet succeeded in producing any gas in quantity which yields this composite spectrum. It is only to be obtained by a mixture of carbon monoxide with cyanogen.

To obtain the heavier gases krypton and xenon, a large amount of air was allowed to evaporate quietly; the residue was freed from oxygen and nitrogen, and then consisted of a mixture of krypton, xenon, and argon, the last forming by far the largest portion of the gas; this mixture was liquefied by causing it to flow into a bulb immersed in liquid air, and the bulk of the argon was removed as soon as the temperature rose, the krypton and the xenon being left behind. By many repetitions of this process we were finally successful in separating these three gases from each other. While krypton has a considerable vapour-pressure at the temperature of boiling air, the vapour-pressure of xenon is hardly appreciable, and this afforded a means of finally separating these two gases from one another; in the complete paper the operations necessary to separate them are fully described.

For neon the process of preparation was different. The air liquefier furnished a supply of liquid air; the gas escaping from the liquefier consisted largely of nitrogen; this mixture was liquefied in a bulb immersed in the liquid air which the machine was making. When the bulb had been filled with liquid nitrogen a current of air was

blown through the liquid until some of the gas had evaporated. That gas was collected separately, and deprived of oxygen by passage over red-hot copper; it contained the main portion of the neon and the helium present in the air. The remainder of the nitrogen was added to the liquid air used for cooling the bulb in which the nitrogen was condensed. Having obtained a considerable quantity of this light nitrogen it was purified from that gas in the usual manner, and the argon containing helium and neon was liquefied. By fractional distillation it was possible to remove the greater portion of the helium and neon from this mixture of gases, leaving the argon behind. Many attempts were made to separate the helium from the neon. Among these was fractional solution in oxygen, followed by a systematic diffusion of the two gases; but it was not found possible to raise the density of the neon beyond the number 9.16, and its spectrum still showed helium lines. It was not until liquid hydrogen made by an apparatus designed and built by one of us (M. W. T.), had been produced in quantity, that the separation was effected; the neon was liquefied, or perhaps solidified, at the temperature of boiling hydrogen, while the helium remained gaseous. A few fractionations serve to produce pure neon; we did not attempt to separate the helium in a pure state from this mixture.

That these are all monatomic gases was proved by determination of the ratio of their specific heats by Kundt's method; the physical properties which we have determined are the refractivities, the densities, the compressibilities at two temperatures, and of argon, krypton, and xenon the vapour-pressures and the volumes of the liquids at their boiling points.

The results are shown in the table.

The compressibilities of these gases also show interesting features. They were measured at two temperatures—11.2° and 237.3°: the value of P.V. for an ideal and perfect gas at 11.2° is 17,710 metre-cubic-centimetres, and at 237.3° to 31,800. This is, of course, on the assumption that the product remains constant whatever be the variation in pressure. Now with hydrogen at 11.2° C. the product increases with the rise of pressure; with nitrogen, according to Amagat, it first decreases slightly and then increases slightly. With helium the increase is more rapid than with hydrogen; with argon there is first a considerable decrease, followed at very high pressures by a gentle increase, although the product does not reach the theoretical value at 100 atmospheres pressure; with krypton the change with rise of pressure is a still more marked decrease, and with xenon the decrease is very sudden. At the higher temperature the results are more difficult to interpret; while nitrogen maintains its nearly constant value for P.V., helium decreases rapidly, then increases, and the same peculiarity is to be remarked with the other gases, although they do not give the product of P.V. coinciding with that calculable by assuming that the increase of P.V. is proportional to the rise of absolute temperature.

These last experiments must be taken as merely preliminary; but they show that further research in this direction would be productive of interesting results.

The spectra of these gases have been accurately measured by Mr. E. C. Baly, with a Rowland's grating; the results of his measurements will shortly be published. It may be remarked, however, that the colour of a neon-

* Abstract of a Paper read before the Royal Society, November 15, 1900.

	Helium	Neon	Argon.	Krypton.	Xenon.
Refractivities (Air = 1)	0.1238	0.2345	0.968	1.449	2.364
Densities of gases (O = 16) ..	1.98	9.97	19.96	40.88	64
Boiling-points at 760 m.m. ..	?	?	86.9° abs.	121.33° abs.	163.9° abs.
Critical temperatures	?	Below 68° abs.	155.6° abs.	210.5° abs.	287.7° abs.
Critical pressures	?	?	40.2 metres.	41.24 metres.	43.5 metres.
Vapour-pressure ratio	?	?	0.0350	0.0467	0.0675
Weight of 1 c.c. of liquid	?	?	1.212 grms.	2.155 grms.	3.32 grms.
Molecular volumes	?	?	32.92	37.84	36.40

tube is extremely brilliant, and of an orange-pink hue; it resembles nothing so much as a flame, and it is characterised by a multitude of intense orange and yellow lines; that of krypton is pale violet, and that of xenon is sky-blue. The paper contains plates showing the most brilliant lines of the visible spectrum.

That the gases form a series in the periodic table, between that of fluorine and that of sodium, is proved by three lines of argument:—

- (1). The ratio between their specific heats at constant pressure and constant volume is 1.66.
- (2). If the densities be regarded as identical with the atomic weights, as in the case with diatomic gases such as hydrogen, oxygen, and nitrogen, there is no place for these elements in the periodic table. The group of elements which includes them is:—

Hydrogen.	Helium.	Lithium.	Beryllium.
1	4	7	9
Fluorine.	Neon.	Sodium.	Magnesium.
18	20	23	24
Chlorine.	Argon.	Potassium.	Calcium.
35.5	40	39	40
Bromine.	Krypton.	Rubidium.	Strontium.
80	82	85	87
Iodine.	Xenon.	Cesium.	Barium.
127	128	133	137

(For arguments in favour of placing hydrogen at the head of the fluorine group of elements, see Orme Masson, *CHEMICAL NEWS*, 1896, vol. lxxiii., p. 283).

- (3). These elements exhibit gradations in properties such as refractive index, atomic volume, melting-point, and boiling-point, which find a fitting place on diagrams showing such periodic relations. Some of these diagrams are reproduced in the original paper. Thus the refractive equivalents are found at the lower apices of the descending curves; the atomic volumes, on the ascending branches, in appropriate positions; and the melting- and boiling-points, like the refractivities, occupy positions at the lower apices.

Although, however, such regularity is to be noticed, similar to that which is found with other elements, we had entertained hopes that the simple nature of the molecules of the inactive gases might have thrown light on the puzzling incongruities of the periodic table. That hope has been disappointed. We have not been able to predict accurately any one of the properties of one of these gases from a knowledge of those of the others; an approximate guess is all that can be made. The conundrum of the periodic table has yet to be solved.

THE ANALYSIS OF URANIUM AND VANADIUM ORES.

By OLIVER P. FRITCHLE.

THIS scheme for the determination of uranium and vanadium is particularly adapted to the mineral carnotite, which is principally a hydrous potassium uranium vanadate. Both uranium and vanadium can be reduced to their lower oxides in a sulphuric acid solution, with metallic aluminum, and then again oxidised to their higher oxides with a solution of potassium permanganate.

Weigh into a 6-oz. flask $\frac{1}{2}$ gm. of the finely powdered ore, moisten with a few drops of water, add 10 c.c. of nitric acid, and digest at a slow boiling temperature for about one hour. Remove from the source of heat, dilute with 10 c.c. of water, add gradually to neutralisation a saturated solution of Na_2CO_3 , and then 5 c.c. excess; then add 20 c.c. of a 20 per cent solution of NaOH . Boil slowly for about half an hour, and then allow the precipitate to settle until the supernatant liquid is clear.

The uranium, vanadium, and iron are all precipitated by

the sodium carbonate, but on adding a moderate excess of sodium carbonate and a large excess of NaOH , the vanadium is dissolved, while the uranium and iron remain insoluble. Uranium is easily precipitated by sodium carbonate and sodium hydrate in the presence of an iron salt.

Filter off the precipitates of iron and uranium, and wash a few times with a solution of NaOH . Do not wash with water, as it washes the uranium precipitate through the filter. The filtrate should show no uranium on testing a nitric acid solution with potassium ferrocyanide. The filtrate contains all of the vanadium, and it could be determined from this solution were it not for the fact that so large a volume of salt solution is difficult to evaporate, and displace with sulphuric acid without violent bumping and considerable loss by spattering. Furthermore, V_2O_5 is not easily soluble in nitric acid, and only by long digestion is all of the vanadium obtained in solution. Therefore it is more expedient to determine the vanadium in a separate sample as given below.

Dissolve the precipitates on the filter with 20 c.c. of hot dilute nitric (1:1), letting the solution run into the original flask. Dilute the solution with about 40 c.c. of water, add NH_4OH until a slight permanent precipitate forms; then add 40 c.c. of a saturated solution of ammonium carbonate ($\text{NH}_4)_2\text{CO}_3$, freshly prepared and heated slightly to remove any excess of CO_2 . Uranium is easily soluble in an excess of ammonium carbonate, and the iron is insoluble. Heat to boiling a few minutes, but do not boil, as the ammonium carbonate will be decomposed, and the uranium precipitated by the ammonia. Filter off the iron precipitate, and wash well with a 2 per cent solution of ammonium carbonate. The filtrate contains all of the uranium, and should not show a trace of vanadium by testing with hydrogen peroxide in a nitric acid solution. A red colouration shows the presence of vanadium. Add slowly 20 c.c. of dilute sulphuric (1:1), and boil until dense white fumes are given off. Cool, dilute to about 100 c.c., add about 4 sq. in. of sheet aluminum, cut into half a dozen stripes, and boil for half an hour or until the yellow solution is reduced to a sea-green colour. Fill the flask with water, decant into a No. 4 beaker, and titrate the uranous solution, hot, with a standard solution of potassium permanganate. The iron equivalent of the permanganate, multiplied by 120/56, will give the amount of uranium in the $\frac{1}{2}$ gm. sample.

Dissolve the iron precipitate with hot dilute HCl , add 10 c.c. sulphuric acid, boil to white fumes, cool, dilute, reduce with metallic aluminum, dilute, decant into a beaker, and titrate with the standard solution of potassium permanganate.

The vanadium is determined in a separate sample as follows:—Weigh into a 6-oz. flask $\frac{1}{2}$ gm. of the ore, add a few drops of water, 10 c.c. nitric acid, and 10 c.c. sulphuric acid. Boil until the nitric acid is all expelled, and dense white fumes of sulphuric acid are evolved. Cool, dilute, add aluminum, and reduce the uranium, vanadium, and iron by boiling for half an hour. Fill the flask with water, decant into a beaker, and titrate, hot, with the standard solution of potassium permanganate. The number of c.c. of permanganate, less the number of c.c. required for the uranium and iron, is the permanganate required to oxidise the vanadium, which, multiplied by $51.7/112$, gives the amount of vanadium in the $\frac{1}{2}$ gm. sample.

The end reaction of vanadium is slow, and permanganate must be added slowly until a permanent pink is obtained.

I prefer the use of aluminum to either zinc or sulphurous acid as a reductor. In the case of vanadium, if sulphurous acid is used, the vanadium is not reduced as low as when aluminum is used, being reduced only to the blue colour, and requiring, approximately, half the amount of potassium permanganate.

The volumetric estimation of vanadium by potassium permanganate is a beautiful titration, changing from purple through blue, green, and yellow to pink.—*The Engineering and Mining Journal*, November 10, 1900.

A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART V.

By HARRY BREARLEY.

(Concluded from p. 247).

SILICON (continued).

II. THE ESTIMATION OF SILICA IN REFRACTORY MATERIALS.

715. CAMERON (C. N., lxi., 171).—After fusion all evaporations should be made with HCl not H_2SO_4 . One evaporation does not make all the SiO_2 insoluble. The influence exerted by Fe, Al, Ca, and organic matter is observed.
716. CRAIG (C. N., lx., 227).—On account of imperfect dehydration of SiO_2 and contamination of precipitates with soda salts, fusion is undesirable. Drive off SiO_2 with HF and H_2SO_4 . A simple apparatus for purifying HF. (See 736).
717. ARCHBUTT (J. S. C. I., 1892, 215).—Confirms 716. The SiO_2 of clays is not completely insoluble after $KHSO_4$ fusion. Filtrate evaporated with H_2SO_4 .
718. ROCHOLL (C. N., xli., 234).—Limestones, siliceous ores, and like minerals are readily decomposable with HCl after strong ignition; the bases already present acting as flux.
719. KERN (C. N., xxxv., 203).—A process for the estimation of SiO_2 , Fe_2O_3 , Al_2O_3 , MnO, CaO, and MgO in fire-clay, &c. Opens out by Na_2CO_3 fusion.
720. ANON. (C. N., v., 228).—Comparative value of fire-bricks determined by heating simultaneously under layer of limestone and observing the degree to which they have been acted upon.

III. THE ESTIMATION OF SILICA IN SLAGS AND SILICATES.

721. ROSE (C. N., i., 179).—Silicates may be decomposed by heating with powdered ammonium fluoride. Also HOFFMAN (C. N., xvii., 94).
722. AVERY (C. N., xix., 270).—Almost any acid with a normal fluoride will completely dissolve silicates; the loss of SiO_2 need only be very slight.
723. KUHLMANN (C. N., xi., 194).—Silicates decomposed in current of HF at dull redness. Necessary apparatus described.
724. STORY-MASKELYNE (C. N., xxi., 27).—Decompose silicate in a special form of platinum apparatus, collect volatile SiO_2 in ammonia, precipitate the neutral solution with KCl and alcohol, and weigh as fluo-silicate.
725. ALLEN (J. C. S., lxx., 575).—A small crucible containing the powdered silicate moistened with H_2SO_4 is placed in a larger one containing $H_2SO_4 + HF$. A dish of cold water placed over the larger crucible causes re-distilled HF to drop into the smaller.
726. FELLEBERG (C. N., xiv., 243).—Fuse with $CaCl_2 + CaO$, leach with water; the solution contains only Ca and the alkalis.
727. GLINKA (J. C. S., lxiv., ii., 491).—Fuse with $CaCO_3$, decompose melt with nitro-hydrochloric acid, and proceed as usual.
728. BONG (C. N., xxxvii., 112).—Silicates are completely decomposed by fusion with Pb_3O_4 at low temperatures. Dissolve melt in HNO_3 , and evaporate for SiO_2 .
729. JANNASCH (C. N., lxxii., 51).—As Bong, but uses lead carbonate.
730. LECLERC (C. N., lxxvii., 27).—As Bong, but does not appear to evaporate before filtering off the "completely insoluble hydrated silica."
731. HEMPEL (C. N., xlv., 81).—Fuse silicate with sub-nitrate of bismuth, dissolve in HCl, and evaporate for SiO_2 .

732. CHATARD (C. N., i., 279).—As Hempel, but uses the oxide.
733. ILES (C. N., xliii., 78).—Decompose by fusion with KHO in Ag crucible. The SiO_2 is washed with ammonia to remove AgCl. (See 219).
734. HOLTHOF (C. N., li., 18).—Fuse with $NaHCO_3$. At the moment of formation of the normal carbonate the base has an intensified affinity for SiO_2 .
735. SHIMER (C. N., lxx., 102).—Spinel (magnesium aluminate), which may occur in blast-furnace slags, is not decomposed by alkaline fusion nor with $HF + H_2SO_4$; it is by $KHSO_4$. (See 741).
736. GILBERT (C. N., lxi., 270 and 281).—Evaporation of fusion with HCl does not make all the SiO_2 insoluble. $CaCl_2$ assists dehydration, but neither CaO nor Al_2O_3 tend to re-form silicates at higher temperatures; MgO does. (See 757).
737. LINDO (C. N., i., 25).—The character of the SiO_2 may be vitreous or amorphous, according to the varying treatment of the alkaline melt with acids.
738. ILES (C. N., i., 194).—Hot slags plunged into cold water are completely decomposed by HCl. Complete analysis of slags; lime is determined in the presence of the iron.
739. JONES (J. I. S. I., 1888, i., 383).—Compares the fusion process and dissolving of chilled (738) samples in HCl. Dehydrates SiO_2 in either case at $120^\circ C$.
740. CAMP (J. I. S. I., 1897, i., 578).—Decompose blast-furnace slags with HCl, and evaporate twice for SiO_2 .
741. MEEKER (J. S. C. I., 1897, 636).— H_2SO_4 dehydrates better than HCl evaporation, but the filtrate cannot be used for the estimation of Ca and Al. Yields good results in the presence of spinel (735).
742. JANNASCH (J. C. S., lxx., 576).—Fuse with boric acid. Boric acid eliminated by evaporation with HCl and methyl alcohol.
743. JANNASCH (J. C. S., lx., 619).—Decomposed by heating with HCl in sealed tubes.
744. WINKLER (C. N., i., 215).—Pyrogenous silicates in fine powder are dissolved by alcohol saturated with HCl.
745. HUTCHINGS (C. N., liv., 173).—A neat qualitative analysis of silicates, chiefly by means of the blow-pipe.
746. FRIEDBURG (C. N., lxii., 22).—A complete analysis (Si, Fe, Al, Mn, Ca, Mg, Ba, Sr, alkalis, H_2O , P, F, and Ti) of insoluble silicates.
747. HILLEBRAND (C. N., lxxviii., 43).—A lengthy review of the analysis of silicate rocks, paying much attention to the elements occurring in small amounts.

IV. MISCELLANEOUS.

748. BARROWS and TURNER (J. C. S., lxi., 551).—Volatilisation of the iron with Cl is not suited to the estimation of slags containing iron compounds; the sample should be decomposed by cupra-sodium chloride. (See 673 and 694).
749. THOMAS (J. I. S. I., 1888, ii., 241).—The percentage of Si in a pig varies; it is higher at the point of a pig than at the butt.
750. PHIPSON (C. N., xi., 278).—Si exists in iron in two states, distinguished by their behaviour with aqua regia.
751. TOSH (C. N., xliii., 145, 178, and 217).—Discredits the existence of Phipson's graphitic silicon, and also the later statement that the two forms are the silicide and silicate.
752. HOGG (J. S. C. I., 1895, 245 and 262).—Is Si capable of existing in two conditions analogous to that of C in the hardened and annealed steels?
753. MORTON (C. N., xxix., 107).—Experiments showing that Si exists in pigs as a silicide of iron. Also JORDON and TURNER (J. C. S., xlix., 215).

754. CARMOT and GOUTAL (Y. C. S., lxxii., ii., 555).—Si exists in iron as silicides intermediate between Fe_3Si_2 and Fe_2Si_3 , but it combines with Mn in preference to iron.
755. SEKY (C. N., xvi., 187).— SiO_2 thrown down by evaporation with HCl can retain small amounts of P.
756. HAUTEFEUILLE (C. N., i., 272).— SiO_2 should not be dehydrated in the presence of phosphoric acid above 100°C .
757. LORD ("Metall. Anal.," p. 13).—In the presence of CaO , dehydrate SiO_2 at 100°C ; higher temperatures may cause the SiO_2 to re-combine with the bases. (See 736).
758. LAUFER (C. N., xxvii., 162).—By melting a silicate with a salt of P, the metallic oxides are dissolved, but not the quartz; separate amorphous SiO_2 with NaHO .
759. LUNGE (Y. S. C. I., 1897, 762).—Aqueous soda attacks quartz; Na_2CO_3 is better for separating amorphous silica.
760. MICHAELIS (Y. S. C. I., 1895, 1065).—Boiling 10 per cent Na_2O does not attack quartz. Quartz in clay is often so finely divided as to pass through filter-paper.
761. CARON (C. N., v., 102).—The only acids which attack crystallised silicon are nitro-fluoric.
- 761a. NEWTH (Y. S. C. I., 1895, 1074).—Crystallised Si burns in HF gas at a moderate temperature.
762. FRESSENIUS and HINTZ (C. N., ix., 85).—Cryolite is analysed by heating with H_2SO_4 in a leaden tube; the volatile fluorides are absorbed in ammonia. For other means of estimating Si when F is present see Hampe (Y. C. S., lxii., 1127), Reich (Y. C. S., lxx., 531), and Stahl (Y. C. S., lxx., 621).
763. HIRSCHWALD (C. N., lxii., 24).—Microcosmic fusion is of doubtful analytical significance, because SiO_2 is appreciably soluble in the head of the double salt.

THE PLASTICITY OF SOLID BODIES, AND ITS CONNECTION WITH THE FORMATION OF ROCKS.

By W. SPRING.

(Concluded from page 249).

It often happens that the volume of the product of combination of two or more bodies is less than the sum of the volumes of the uncombined elements. For example, the formation of sulphide of silver is accompanied by a contraction of 6.3 per cent of the volume of its elements. The contrary occurs but rarely; for example, the hydrated sulphide of arsenic is 4.8 per cent greater in volume than the sum of the water and of the anhydrous trisulphide of arsenic. Now, if we compress a mixture of elements in a state to give a compound of the first type, we notice that pressure assists the chemical reaction the more, as the reciprocal solubility of the two elements is the more pronounced. For example, silver and sulphur combine well under pressure: they also combine when they are heated together. On the other hand, zinc and sulphur—which may be melted together without, so to speak, forming sulphide of zinc—hardly combine any better under pressure, while the contraction resulting from the combination amounts to nearly 5 per cent of the volume of the elements. We have made a large number of compounds of the first type with the aid of compression, especially sulphides and arsenides. The old chemical adage, *Corpora non agunt nisi fluida*, can no longer therefore be taken as literally true; it is the speed of the reaction which depends principally on the fluidity of the substance, but not the faculty of reacting.

It is easy to foresee what will be the effect of compression on compounds of the second type; there is not only no trace of reaction, but the compound is decomposed. The hydrated sulphide of arsenic is resolved into anhydrous sulphide and water; cupro-calcic acetate, which is more voluminous than its components, gives acetate of copper, acetate of calcium, and water.

In the same way several substances in an allotropic state have been changed into a more dense form; for instance, prismatic sulphur was easily converted into the octahedric variety, and amorphous arsenic became crystalline. These facts received a remarkable confirmation when M. Moissan showed that if the crystallisation of carbon dissolved in melted iron takes place under sufficient pressure, small diamonds are formed. There is another fact connected with these results; experiment has shown that compression only produces a permanent diminution in the volume of a solid body, when this latter allows of the formation of a more dense allotropic condition. When this condition does not exist, compression only diminishes the volume of the solid during its application. Thus solids behave in the same way as liquids and gases, and have as perfect an elasticity, and there is nothing, moreover, which authorises the opinion that one chemical element can be transformed into another denser one by mechanical means.

All these results show why compression alone of sedimentary deposits has not been able to cause the solidification of rocks. But Nature has probably put into force a factor of which we have not yet taken notice, namely, moisture. We know that the solubility of many bodies in water is increased by pressure; may we not ask whether bodies apparently insoluble, like sand, may not give some signs of dissolution when strongly compressed in contact with water. We have therefore made experiments in this direction, the results of which may be summed up as follows:—In the same manner as with compound bodies, the volume of a solution is hardly ever the exact sum of the volume of the solvent and of the body dissolved; as a rule it is less. Now all bodies which fulfil this condition unite incomparably better in a moist state than in a dry state; the reason is that their solubility increases with pressure, the solvent becomes charged with matter, and forms a supersaturated solution at the ordinary pressure. When the pressure ceases, the excess of matter dissolved is deposited on the remainder of the solid and cements the grains together; it is, in fact, a true *setting*, such as occurs with plaster. On the other hand, bodies, of which the solution is accompanied by an increase of volume, unite with difficulty when moist under pressure, because the solvent re-dissolves the material when the pressure ceases.

Again, bodies which are generally considered to be insoluble in water, such as hydrate of iron, peroxide of manganese, various carbonates, &c., join better when wet than dry. We have obtained agglomerations the surface of which presented an extremely interesting appearance, being vitreous and transparent, and seeming to point to the commencement of liquefaction. Carbonate of copper showed this to a remarkable degree; though the interior of the block preserved the pale green colour of the powder, the surface seemed to be covered with a layer of malachite, green and transparent. It recalled the slipping surfaces of our older rocks. We may therefore suppose that the grains of sand or gravel, in "banket" for example, become covered with a supersaturated solution of silicic acid by the action of pressure, and that this solution in unstable equilibrium has furnished the cement necessary for solidification. It is not without interest to remark that there really exists between the grains of sand and gravel in banket, a siliceous layer which escapes general notice. Silicic acid, however, even in the solid state, has the property of dissolving in a solution of potash or soda, while the quartzose grains are refractory and are only attacked with extreme slowness. A block of banket should therefore become disintegrated in an alkaline

solution. Experiment confirms this deduction; conglomerates of recent formation required only a few weeks to disintegrate at the temperature of boiling water; older rock formations offered a greater resistance to alkalis. They only became friable, while the former was reduced to sand. The reason of this greater resistance may be found in the fact that the silicic acid has, in the course of time, passed more completely into the quartzose state which is refractory to potash.

We must not, however, think that these last observations solve the problem. There are, in fact, some compact rocks which have never been subjected to enormous pressures, that is, anything approaching 70 tons. It is therefore necessary to try whether the simple infiltration of a silicic solution can cement the particles through the slow evaporation of the water of solution. For this purpose we tried to cement sand together by means of a solution of silicic acid. We know that a solution of this acid submitted to slow, spontaneous desiccation, forms a vitreous mass of great hardness. We therefore made a paste of sand and silicic acid, and allowed it to dry. There was not a trace of agglutination. The reason of this discouraging result was seen at once on making a microscopic examination of the substance. The silicic acid had, in fact, adhered to the grains of sand, but, owing to the enormous contraction which accompanied the desiccation, it cracked in every direction round the grains. The cause of the failure being known, its remedy was apparent. By the application of a slight but continuous pressure we obtained a commencement of solidification. If the result was not entirely satisfactory, at any rate it proved the possibility of obtaining perfect results, by suitably varying the conditions and allowing time to play a more important part.

The solidification of rocks may then have taken place in Nature, after the infiltration of siliceous waters accompanied by a compression of relatively low intensity, but lasting a long while. The co-operation of these factors seems to be indispensable, as pressure alone, like infiltration alone, is inefficacious, as far as one may conclude from laboratory experiments.

The solidification of calcareous rocks may also be explained by the above process; the *débris* of shells impregnated with a solution of acid calcic carbonate, has become cemented together on the escape of the carbonic acid, by diffusion into the atmosphere, and by the slow crystallisation of the dissolved calcareous matter, in greater proportion as the pressure was increased.

Finally, even if the phenomenon of regelation, mentioned at the commencement of this article, is not confined to ice, it has not, at any rate, played a part in the formation of rocks in any way approaching that which it played in the history of glaciers.—*Revue Générale des Sciences*, Sept. 30, 1900.

A NEW REACTION FOR THE DETECTION AND ESTIMATION OF SMALL QUANTITIES OF NITROUS ACID.

By H. ERDMANN.

It has been known for the last twelve years that the addition of mineral acids to cultures of the cholera bacillus gives rise to the formation of a violet colouration (Brieger, *Deutsche Medicinische Wochenschrift*, 1887, 303 and 469). Bujvid, who has examined this reaction in a more detailed manner, has found that it is due to the formation of coloured salts with a characteristic base. This latter can be isolated in the form of reddish-brown flakes by shaking the violet solutions, previously made alkaline, with benzene.

By dissolving this "cholera-red" in concentrated sulphuric acid, and then pouring the solution into dilute

soda, it is easily transformed into a new colouring base, "cholera-blue." The first of these substances is converted into indol by distillation with powdered zinc. E. Salkowski (*Virchow's Archiv*, cx., 366), has shown that the choleraic bacteria are constantly producing nitrous acid, and that the phenomena of colouration observed in cultures of these bacilli is nothing else than a known reaction of nitrous acid (Wencki, *Berichte*, viii., 727).

It has been recognised later on that this latter is a product elaborated by the numerous bacteria belonging especially to the series of pathogenic anaerobes. It follows from this observation that the presence of nitrous acid in certain media, particularly in potable waters, should be considered as an indication of the presence of such and such bacteria, if it is desirable to form an opinion on the spot without the aid of any special apparatus; the above mentioned method, however, still leaves much to be desired.

In his experiments on spring or well-waters used for drinking purposes by either man or beast, the author takes into consideration quantities of nitrite which, on an average, correspond to a normal solution of one millionth, so that a solution of 100,000th would indicate a microbial activity of considerable power. It is only under very exceptional circumstances that the bacillary production of nitrite would lead to a concentration corresponding to a normal solution of a 10,000th; he has therefore taken no notice of solutions of less strength than 1 centigram. of nitrous acid per cubic metre. For proportion varying from 1 centigram. to 1 grm. per cubic metre, it is indispensable to make a quantitative estimation, at least approximately, if we wish to know whether the water under examination must be condemned, or looked upon with more or less suspicion.

It is easy to see how insufficient are the usual methods used for this purpose by referring to the words of Fr. Brismann. (Lunge, *Untersuchungsmethoden*, i., 728), in the last edition of Boeckmann's book:—"It is rarely necessary to estimate the nitrous acid in the analysis of waters used for drinking or domestic purposes. A really pure water should not contain any nitrous acid, and those which contain more than mere traces must be looked upon as containing impurities of such a nature as to render them unfit for drinking."

It is therefore interesting from the practical point of view to determine the quantitative value of these "traces" of nitrite. The appearance of certain waters which are dangerous, both for man and beast, is often excellent, even when their proportion of nitrite has reached the upper limit (1-2 grms. of nitrous acid per cubic metre). On the other hand, it is not permissible to establish the character of water, as is often done, on its richness in chlorine, as the salt is often derived from mineral or saline deposits of considerable extent.

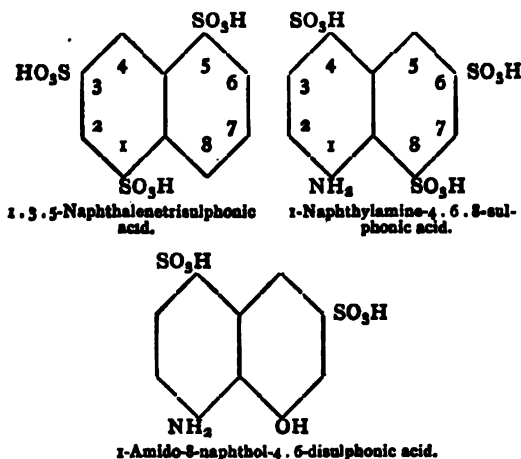
If we can succeed in transforming the nitrous acid of a water integrally into a strongly coloured azoic compound, it would be possible to base an examination at once qualitative and quantitative on the intensity of the colouration obtained. (See Annahelm, *Berichte*, ix., 1351).

It is necessary to avoid the use of phenylenediamines (see Griess, *Berichte*, xi., 624), and of analogous bodies which give insufficiently characteristic brown colourations. These compounds are, further, too sensitive to the action of oxidising agents, with which they give rise to colouration phenomena analogous to those which are caused by nitrous acid under the same conditions.*

As a result of numerous preliminary experiments the author has decided to use 1.8-amino-naphthol-4.6-disul-

* For the same reason the use of the starch-iodine method cannot be allowed (see Kaemmerer, *Zeit. f. Analyt. Chem.*, 377), which is unfortunately too often used in bacteriological laboratories. In the present state of the water question, we must take notice of the possible presence, not only of oxide of iron, but also of free chlorine, ozone, and other oxidising agents. When submitted to the action of iodide of starch, a water which had been completely disinfected by one of these substances, would behave in such a manner as to be looked upon with suspicion.

phonic acid obtained from 1.3.5-naphthalene-trisulphonic acid (*Berichte*, xxii., 3186), by nitration, reduction, and treating with boiling caustic soda (*Ch. Ind.*, 1898, p. 523; *Lehne's Färbertg.*, x., 358).



This acid has the property of combining very easily with the diazotic derivatives in acid solution, giving rise to monoazoic colouring materials which, by their solubility, great tinctorial power, and characteristic colour, are perfectly adapted for the object it is desired to attain. The operation is carried out in the following manner:—

50 c.c. of the water to be examined are mixed with 5 c.c. of a hydrochloric solution of sulphanilic acid (2 grms. of sulphanilate of soda per litre), and after the lapse of ten minutes, as the dinitration does not take place instantaneously in very dilute solutions, about 0.5 grm. of 1-amino-8-naphthol-4,6-disulphonic acid is added in the solid state, and in the form of an alkaline salt.

In the presence of nitrous acid a wine-red colour is produced which reaches its maximum intensity in about an hour. For quantitative estimations the shade obtained is compared with a series of tubes of known strength prepared at the same time.

For this purpose we make use of normal solutions of nitrite at a millionth, one hundred-thousandth, and one ten-thousandth. When we have to make really exact measurements, these solutions must be prepared at the last moment by diluting the normal solution, as, in such dilute solutions, there soon develop micro-organisms, whose activity would gradually diminish the proportion of nitrous acid present. It is for this reason that a diminution takes place in the quantity of nitrous acid in impure waters which have been kept too long. However, a spring water, which had been the cause of an outbreak of malignant pustule, still gave a distinct colouration with the reagent in question, after the lapse of a year. It must therefore be admitted that nitrous acid can be detected long after the disappearance of the micro-organisms by which it was produced. This property gives the chemical examination of water a distinct advantage over the bacteriological method, by which, on account of the ephemeral life of anaerobic bacteria in water, we are rarely able to isolate them in an effective manner.

Among the methods of investigation previously known that of Griess (*Berichte*, xii., 427), must be mentioned; it is based on the use of α -naphthylamine. Although fairly sensitive this method is not very applicable to the object we have in view. In the first place, the reagent used is rarely colourless, and it is difficult to keep. Another drawback is met with in the slight solubility of the yellow-red colouring material, which in the examination of hard or saline waters, is deposited in a flocculent state. It follows that this phenomenon seriously interferes with quantitative estimations. The author cannot give an

opinion on the use of this reagent for the detection of nitrosylsulphuric acid in sulphuric acid (Lunge and Luoff, *Zeits. f. Angewandte Chem.*, 1894, 348), as he has never made any experiments on the subject. But as far as concerns the analysis of waters, he considers it insufficiently exact and characteristic. Riegler's method (*Zeits. f. Anal. Chem.*, xxxv., 677, 377; Koenig, *Untersuchenland. u. Gew. Wichtiger Stoffe*, Second Edition, 1898, 610), is even less able to bear comparison with the new method; in fact, naphthionic acid, like many other easily diazotable substances which the author has experimented with, possesses a fluorescence which interferes disadvantageously with the reaction.

Solutions of 1-amino-8-naphthol-4,6-disulphonic acid are not at all dichroic under the existing conditions. Oxidising agents, such as perchloride of iron, cause the formation of a yellow colouration. It is impossible, however, to confound this latter with the bright wine-red shade on which the new method depends.

The Laboratory of Applied Chemistry, "Halle-a-S., Domplatz, 1," will supply samples of the new reagent ready for use, gratuitously to those interested in the matter. Instead of using solutions of nitrite of known strength as standards of comparison, a coloured paper scale paper may be used for colorimetric comparison.—*Berichte*, vol. xxxiii., p. 210.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 251).

Dilatometer Experiments (continued).

Experiment 30.—A continuation of Expt. 29. Starting from vitreous selenium, the temperature was rapidly raised, and in this case attained almost 175° before the transformation began. The subsequent readings are as follows:—

Temp.	Scale.	Time.
175°	390	9 : 39
186	397	9 : 40
195	395	9 : 41
203	405	9 : 47
199	393	11 : 20

This confirms the results of Experiment 29.

The tube was next heated to 225° to effect fusion, and then cooled to 125° to observe the nature of the transformation at that point. The readings are:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
232°	560	11 : 45	176°	310	12 : 43
125	265	12 : 03	—	311	1 : 01
127	231	12 : 16	198	377	1 : 30
—	177	12 : 32	206	399	1 : 50
—	177	12 : 37			

The transformation at 125° brings the dilatometer back to a reading almost identical with that obtained by interpolation from the results of Experiment 28, where the dilatometer contained selenium which had been changed over at 200° and then cooled down.

In the experiments which follow, special care was taken to eliminate water from the system. The paraffin used in all these later experiments was dried by sodium. For drying selenium which has been precipitated out of an aqueous solution of selenious acid, it is by no means sufficient to heat it for an hour or two at 120°, although by this means it goes over to the metallic form, and one would suppose that such a change would be likely to set free all the moisture present; on fusing such a preparation in a tube, however, a very considerable quantity of water is still given off. The material here used was dried by heating to 330° for some time in an exhausted A-shaped

* From the *Journal of Physical Chemistry*, vol. iv.

tube, containing sulphuric acid in the other arm. By observing these precautions, and by occasionally opening the dilatometer tube to permit the escape of the small bubbles which had developed in the bulb, it was possible to get very concordant results. These experiments confirm the earlier ones.

Experiment 32.—New dilatometer.* Starting from metallic selenium, the following observations were made:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
84°	51		223°5	562	3 : 32
153	243		200	477	3 : 36
196	361	2 : 51	192	428	3 : 46
204	386	3 : 06	198	374	3 : 59
219	430	3 : 12	196	362	4 : 11
228	525	3 : 19			

Here the last reading at 196° accords almost exactly with the first. The material was again fused and then rapidly cooled; on heating up again, the transformation began at about 130° and gave, after coming to rest at 191°, a reading of 355, which accords very closely with the above value of 362 at 196°.

Experiment 36 was made with a new dilatometer. The level at 199° corresponding to the more dense form, was 321 on the scale. After fusion and cooling, it was placed in a bath at 200°; the transformation took place very rapidly, and the level came to 322 at 199°, from which there was no further change during five minutes. The material was again fused and cooled, and then placed in a bath of boiling water until the change was almost completed; it was then replaced in a 200° bath, where it gave a reading of 314 at 197°. The selenium was again fused and cooled down. This time it was placed in a cold bath, whose temperature was then rapidly raised—this being the method by which Siemens prepared his light-sensitive selenium cells. The change began at about 120° and the temperature, after reaching 200°, was maintained for some time at that point; the dilatometer gave a final reading of 321° at 200°. On being transferred to a bath of boiling water, it gave a reading of 36 at 99°. The material was once more fused and then placed directly in the water-bath; the final reading was 44, and as the change proceeds very slowly towards the end, this may reasonably be taken as a trifle too high. Finally, the temperature was raised to 200° and then lowered to 80°; all the readings so obtained were found to lie along one line, and there was nowhere any sign of a change. This line cuts 100° at a little above 40, and 200° a little above 320°.

In order to make sure that the denser form which plays so important a part in these dilatometer experiments was really metallic selenium, the material was removed from this last bulb and its specific gravity determined; the value found was 4.77.

Experiment 36 shows that metallic selenium is the only form which results from the transformation of the vitreous modification, whether at 100° or in a bath whose temperature is rapidly raised to 200°. The readings obtained at 200° are the same in each of these two cases, and are identical with that obtained by holding fused selenium at 200° until the transformation is complete.

Experiment 41.—This was a further attempt to get some indication of a reverse change from the grey metallic into a less dense form, below 100°. Starting from vitreous selenium, the following readings were taken:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
59°	114	11 : 15	80°	134	1 : 20
62	118	11 : 18	81	96	2 : 30
68	130	11 : 27	—	66	4 : 06
74	141	11 : 40	70	47	4 : 15
81	154	12 : 16	69°5	45	4 : 35
83	157	12 : 25	98	92	4 : 45
82	153	12 : 35	—	84	5 : 05

* In the later experiments, a side tube was set on, at an angle, to the bulb of the dilatometer; this facilitates the filling, and can be sealed off afterwards.

The experiment shows that when the change has proceeded some distance at 81°, and the temperature is lowered to 70°, there is no sign of a recovery during twenty minutes, although the last two readings show that the transformation was not complete, *i.e.*, that two forms were present.

Experiment 43.—Confirmatory of Experiment 41. This determination, which was carried on in a dilatometer filled with quinoline, yielded the following readings:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
81°	112	2 : 08	70°	42	4 : 15
—	93°5	2 : 15	69°5	41	4 : 35
—	74	2 : 40	98	71	4 : 48
—	56	3 : 36	—	66	5 : 05
—	52°5	4 : 06			

Experiment 45.—Carried on with the same dilatometer that was used in Experiment 41. Starting from vitreous selenium, these readings were made:—

Temp.	Scale.	Time.	Temp.	Scale.	Time.
65°	127	12 : 32	78°5	150°5	2 : 03
71°5	139	1 : 10	80	152	2 : 16
74	143	1 : 32	81°5	152	2 : 34
77	148°5	1 : 51	78	139	3 : 07

showing that the transformation began at about 80° in this case. The temperature was then raised—

Temp.	Scale.	Time.
107°	178	3 : 36
125	150	3 : 38
130	122	3 : 39
156	158	

Then the dilatometer was placed in the cooler bath—

Temp.	Scale.	Time.
67°5	19	3 : 51
—	17	4 : 15

The dilatometer then stood over night, and registered on the following day—

Temp.	Scale.
58°5	0

Four days later the level was again taken—

Temp.	Scale.	Time.
63°	5	11 : 30
78	28	12 : 40

The bulb then remained at ordinary temperatures for two weeks, sometimes standing for half a day in direct sunlight. At the end of that time a reading was again taken and gave—

Temp.	Scale.
59°	0
69	15

and after standing in sunlight for a few hours more, it gave on the following day—

Temp.	Scale.
70°	17

Two months later a reading was again taken, and gave at—

Temp.	Scale.
63°	4

which shows that no change had taken place.

This experiment illustrates the complete stability of metallic selenium at ordinary temperatures and up to 60°, whether it be kept in diffused daylight or in direct sunlight.

When selenium is heated by itself to 100° it does not always go over entirely to the metallic form. Petersen (1891) found that specimens of selenium which were transformed from the vitreous into the metallic form, at whatever temperature, always contained more or less of the original material. This is illustrated by the following experiment:—

Experiment 46.—A quantity of vitreous selenium was heated for half an hour in a water-bath, and was then

placed in a dilatometer with quinoline. The following are the readings:—

Temp.	Scale.	Time.
74°	103	1:32
80	92	2:16
78	78	3:00
73	70	3:21
67.5	63	3:45

The dilatometer was, later on, heated for five hours between 45° and 60° and showed no signs of a reverse transformation, although raising the temperature afterwards to 80° showed that the transformation had not been entirely completed.

When vitreous selenium is placed in a dilatometer with water, the transformation begins at about 80° and proceeds slowly—but uninterruptedly—to an end, as shown by the following experiment:—

Experiment 69.—3.06 grms. of vitreous selenium were placed in a dilatometer provided with a capillary arm previously calibrated. The following readings were made:—

Temp.	Scale.	Time.
64°	89	11:44
71	98	12:14
77	106	12:30
79	108.5	12:35
81	111	12:40

A slight transformation took place at this last temperature, and the experiment was continued on the following day.

Temp.	Scale.	Time.	Temp.	Scale.	Time.
67°	86		80°	65.5	12:06
82	102.5		79	58.5	12:40
85.5	105	11:05	81	42.5	2:15
84	87	11:28	80	36	4:20
83	81	11:36			

and on the following day—

Temp.	Scale.	Time.
79°	27	11:20
80	26	12:30
—	24	3:30
77	21	5:10

The transformation may be taken as complete.

Calculating the change in density from the difference between the original and final readings at 77°, and assuming the density of vitreous selenium to be 4.3, we find for the density of the metallic form by this experiment 4.77. The determination shows that no other forms make their appearance here but the two mentioned.

In another experiment, also carried on in water, a quantity of selenium was heated in a water-bath for one and one-half hours, after which no further change of volume could be noted. The mass had the familiar steel grey colour. On raising the temperature to 130° no change took place. Some of it was then taken out, and placed in a dilatometer containing aniline, and heated during one and one-half hours to about 150°. This also brought about no further change, showing that in water as well as in other liquids, it is the metallic form which always results from a transformation of the vitreous modification no matter at what temperature this transformation is effected.

(To be continued).

Messrs. F. E. Becker & Co. have just issued a new Illustrated Catalogue of Physical Apparatus, fully illustrating and describing Magnetism, Frictional Electricity, Voltaic Electricity, Electro-magnetism, Electro-plating, and "X" Ray Work, &c. The catalogue is well printed and the illustrations are of good character, and we recommend its perusal to anyone who may be about to fit up or extend a physical laboratory.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

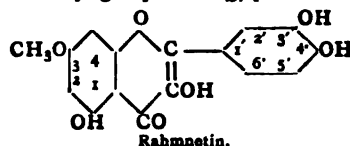
Ordinary Meeting, November 1st, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

(Concluded from p. 254).

139. "Rhamnazin and Rhamnetin." By A. G. PERKIN and J. R. ALLISON.

In rhamnazin (quercetin dimethyl ether) the position of but one methoxyl group is known (*Trans.*, 1897, lxxi., 818), whereas that in rhamnetin (quercetin monomethyl ether) (Herzig, *Monatsh.*, 1888, ix., 548) has not been determined with certainty. On gentle decomposition, rhamnazin, rhamnetin, and quercetin tetramethyl ether give the same phloroglucinol derivative, as in each case the latter reacts with diazobenzene to form a compound, occurring in orange-red needles, m.p. 251–252°. This is *diazobenzene-phloroglucinol monomethyl ether*. (Found C=65.17; H=4.64; N=15.57; CH₃=4.40 per cent); consequently rhamnazin is methoxyrhamnetin, and both contain a methoxyl group in the (3) position.



140. "Luteolin." III. By A. G. PERKIN and L. H. HORSFALL.

On methylation, luteolin (from weld) yields two ethers insoluble in alkali, (a), m. p. 191–192° (*Trans.*, 1896, lxxix., 206), and a more soluble compound, (b), newly isolated, m. p. 161–163°. The latter, C₁₅H₁₀O₅(OCH₃)₃, is the true *luteolin trimethyl ether*, for on decomposition it forms *veratric acid* and *phloroglucinol monomethyl ether* (identified as its diazobenzene derivative, m. p. 251–252°). The ether (a), m. p. 191–192°, appears to be *methyl luteolin trimethyl ether*, due to the existence of a methyl group entering the ring during methylation, or less probably to the presence of methyl-luteolin in weld. On gentle decomposition, *veratric acid*, and a crystalline phloroglucinol derivative result, the latter yielding with diazobenzene, a derivative, in orange-coloured needles, m. p. 198–200°, C₁₅H₁₀O₇(C₆H₅N₂)₂, apparently *diazobenzene-methyl phloroglucinol monomethyl ether*. *Acetyl methyl luteolin*, colourless needles, melts when rapidly heated at 238–240°. Luteolin from the *Genista tinctoria* behaves similarly on methylation. The monopotassium and sodium salts of luteolin have now been crystallised, and have the respective compositions KC₁₅H₉O₆ and NaC₁₅H₉O₆. Weld contains a trace of another colouring matter C₁₅H₁₀O₅, which, on decomposition with alkali, yielded *p-hydroxybenzoic acid*, a trace of *protocatechuic acid*, *phloroglucinol*, and *p-hydroxyacetophenone*. By further reactions, it was identified as *apigenin*. By the gentle action of alkali, morin, a colouring matter of *Morus tinctoria*, yields, in addition to β -resorcylic acid and phloroglucinol (*Trans.*, 1896, lxxix., 697), previously found, some quantity of β -resorcylic aldehyde.

141. "Genistein." II. By A. G. PERKIN and L. H. HORSFALL.

Genistein dimethyl ether, m. p. 137–139° (*Trans.*, 1899, lxxv., 835) yields a monacetyl derivative, m. p. 202–204°, C₁₄H₁₀O₂(OCH₃)₂C₂H₃O, and on gentle decomposition gives *p-methoxyphenylacetic acid* and *phloroglucinol monomethyl ether*, identified as its diazobenzene derivative, m. p. 251–252°. The second product of the methylation, m. p. 200–202° (previously given as 187–189°), appears to be a *methylgenistein dimethyl ether* (compare luteolin). It forms a monacetyl derivative, C₁₇H₁₅O₅C₂H₃O, m. p. 212–214°, and on decomposition gives *methoxyphenyl-*

acetic acid and a phloroglucinol derivative whose diazobenzene compound (probably *diazobenzene methylphloroglucinolmonomethyl ether*), orange-coloured needles, melts at 198–200°. This is identical with that produced from methylulicotrimeric ether. *Genistein diethyl ether*, $C_{14}H_{18}O_5(OEt)_2$, colourless needles, m. p. 132–134°, and its monacetyl derivative, m. p. 168–170°, have been obtained. On decomposition it gives *p*-ethoxyphenylacetic acid and a phloroglucinol compound. These results are in harmony with the constitution of a *trihydroxyphenylketosummaran* previously suggested (*loc. cit.*) for genistein. Genistein from *Genista tinctoria* melts at 291–293°.

142. "The Colouring Matter of the Flowers of *Delphinium consolida*." By A. G. PERKIN and E. J. WILKINSON.

The presence of a yellow colouring matter in these flowers (in the form of glucoside) has been previously notified (*Trans.*, 1898, lxxiii., 275). This compound forms yellow needles, has the composition $C_{15}H_{10}O_6$ (found C=62.85; H=3.63 per cent), and on fusion with alkali gives phloroglucinol and *p*-hydroxybenzoic acid (found C=60.71; H=3.94). The *tetracetyl* compound, $C_{15}H_2O_6(C_2H_5O)_4$ (found C=60.64; H=4.17), is peculiar, for when crystallised from alcohol it melts at about 114–116°, re-solidifies at a higher temperature, and again melts at 181–183°. This is not due to alcohol of crystallisation. The sulphate, $C_{15}H_{10}O_6.H_2SO_4$, orange-red needles (found C=46.71; H=3.39, the *hydriodide*, $C_{15}H_{10}O_6HI$ (found C=44.25; H=3.00, and the *monopotassium salt*, $C_{15}H_9O_6K$ (found K=11.85), have been obtained, but are less stable than is usually the case with such compounds. The colouring matter has properties resembling those assigned to kampherol (Gordin, *Diss. Berns*, 1897), obtained by the decomposition of its methyl ether, kampherid, which exists in galanga root (*Alpinia officinarum*). The investigation will be continued with the hope of ascertaining the identity of this colouring matter with certainty.

143. "Note on Gallinek's Amidomethylnaphthimidazole." By RAPHAEL MELDOLA, F.R.S., and FREDERICK WILLIAM STREETFIELD.

A paper by Gallinek has just been published (*Ber.*, 1900, xxxiii., 2315) on a sulphonic acid of the anhydrosulphonic acid obtained by reducing dinitro- α -acetnaphthalide. Gallinek assumes that the base he has been dealing with is the same as that described by the authors (*Trans.*, 1887, li., 691, and he considers that their failure to separate the base in the free state was due to their having had impure dinitro- α -acetnaphthalide as their raw material. The authors desire to point out that Gallinek is completely in error in his original assumption, and that the base described by him is isomeric, and not identical with that described by the authors in 1887. Gallinek does not state what reducing agent he employed, nor does he refer to the process published by Markfeldt (*Ber.*, 1898, xxi., 1174), but his description of the base as being stable, solid, and crystallisable from water, renders it tolerably certain that he has been investigating Markfeldt's base. Markfeldt also assumed that his base was identical with that described by the authors in 1887. The isomerism was recently made known, and the properties of the base obtained by Markfeldt's process somewhat fully characterised (Meldola and Eynon, *Trans.*, 1900, lxxvii., 1159). The authors think it necessary to indicate the true cause of the apparent discrepancy between the statements of the German investigators and themselves, because they are at present engaged in the further investigation of the nature of the isomerism.

144. "The Amount of Chlorine in Rain Water collected at Cirencester." By EDWARD KINCH.

The author has already published the results (*Trans.*, 1887, li., 92) of the estimation of the chlorine in the water collected in a rain-gauge at the Royal Agricultural College, 443 feet above sea-level, and about 35 miles distant from the sea. The results of his determinations up to the present time are summarised in the table.

The rain-water for periods of six months, namely, summer months, April to September, and winter months, October to March, inclusive, gave the averages shown in the accompanying table.

145. "Researches on the Alkyl-substituted Succinic Acids III. Dissociation Constants." By W. A. BONE and C. H. G. SPARKLING.

The authors have prepared, and determined the dissociation constants of, a number of new alkyl-substituted succinic acids as follows:—

	M. p.	K.
<i>sym</i> Di-isobutylsuccinic	<i>trans</i> 195°	0.0225
	<i>cis</i> .. 97–98	0.056
<i>aa</i> ₁ -Methylpropylsuccinic	<i>trans</i> 158–160	0.0355
	<i>cis</i> .. 92–93	0.0271
<i>aa</i> ₁ -Methylisobutylsuccinic	<i>trans</i> 133	0.0427
	<i>cis</i> .. 88–90	0.0236
<i>aa</i> ₁ -Methylisoamylsuccinic	<i>trans</i> 141–142	0.0236
	<i>cis</i> .. 93	0.0385
<i>aa</i> -Dimethyl- α -ethylsuccinic	139–140	0.0566
<i>aa</i> -Dimethyl- α -propylsuccinic	145	0.060
<i>aa</i> -Dimethyl- α -isopropylsuccinic	141–142	0.0158
<i>aa</i> -Dimethyl- α -isobutylsuccinic	143–144	0.0432
<i>aa</i> -Dimethyl- α -isoamylsuccinic	143–144	0.0616

They then discuss, in the light of the above and previous results, the effect of alkyl-substitution on the magnitude of the dissociation-constant of a succinic acid, and deduce the following conclusions:—

Each alkyl group exerts its own influence upon the constant dependent on its mass and structure. In the case of *normal* radicles, where the influence of "mass" only is to be traced, an increase in the mass of the alkyl is invariably accompanied by a corresponding rise in the constant. In the case of "iso"-radicles there is, however, a "structural" effect opposed to that of mass, except in the case of the *cis* α ₁-substituted acids, which are separately discussed: the magnitude of this structural effect is always dependent on the proximity of the "iso"-linkage to the carbon atom to which the carboxyl group is attached.

146. "The Reaction between Ethyl Alcohol and Hydrochloric Acid." By T. SLATER PRICE, D.Sc.

The present investigation was undertaken in order to measure the velocity of reaction between hydrochloric acid and ethyl alcohol. On re-determining the equilibrium constants it was found that they varied with the amount of acid used. The constants (α) were calculated according to the equation $\alpha = (A-x)(B-x)/(C+x)(D+x)$, where A, B, C, and D are the concentrations of the alcohol, acid, water, and ester respectively at the commencement of the reaction, and x the amount of fresh ester formed when equilibrium is reached. The results obtained agree with those of Zaitsech (*Zeit. Phys. Chem.*, 1897, xxiv., 1), who investigated the action of sulphuric acid on alcohol.

Determinations were made at 77°, 99°, and 129.5°. The value of α decreases as the amount of acid increases: this is well shown especially in the experiments at 77°, as the lower the temperature the greater is the variation.

Periods.	Rainfall in inches.	Chlorine per million.	Equivalent to NaCl, grms. per gallon.	Equivalent to NaCl, lbs. per acre.
Mean of 14 winter periods to March, 1900	14.26	3.55	0.412	19.35
Mean of 14 summer periods to September, 1900	12.78	2.27	0.261	10.40
Average of 14 years to September, 1900	27.04	2.91	0.337	29.75
Mean of 26 winter periods to March, 1900	15.83	3.76	0.435	21.29
Mean of 26 summer periods to September, 1900	14.78	2.58	0.302	14.81
Average of 26 years to September 31st, 1900	30.61	3.17	0.369	36.10

The results obtained by the author are found to agree very well with the equation—

$$dx/d\theta = k_1(A-x)(B-x) - k_2(C+x)(D+x)(B-x),$$

k_1 and k_2 are the velocity constants of the direct and reverse reactions, x is the amount of ester formed after the time θ , and A, B, C, and D have the signification given above. The catalytic effect of the hydrogen ions is assumed to be proportional to the amount $(B-x)$ of acid present, this holding good over a short interval of time, and since both the direct and reverse reactions are affected the factor $(B-x)$ will be extra in each term of the velocity equation. The values of k_1 so obtained, are especially good when no water is present to begin with, that is, when $C=0$ (in no case was ester present at the commencement of the reaction), but when the reaction mixture used contained water to start with, the values of k_1 diminish as θ increases.

The value of k_1 decreases as the concentration of the acid increases, and is also greatly diminished when water is present.

The velocity of reaction increases very rapidly with the temperature, the increase being much greater than in the case of the action of organic acids on alcohol.

PHYSICAL SOCIETY.

Ordinary Meeting, November 23rd, 1900.

Prof. EVERETT, F.R.S., Vice-President, in the Chair.

A PAPER ON "A Self-adjusting Wheatstone's Bridge," by E. H. GRIFFITHS and W. C. D. WHETHAM, was read by Mr. WHETHAM.

The object of this paper is to describe a cheap and easy method of getting a self-adjusting bridge to show on a scale the actual resistance of any wire. Contact with the bridge-wire is made by means of a light horizontal bar which is suspended by a phosphor-bronze strip from the coil of the d'Arsonval galvanometer used with the instrument. A second bar, parallel to and above the first, is rigidly connected with the coil. A wooden beam, worked by clockwork, moves up and down between the bars, and clamps them alternately. When the beam is down, contact is made with the bridge-wire. If this contact is not at the zero-point, a current will flow through the coil, and, if the cell is connected up the proper way, it will turn the coil so as to bring the upper bar nearer to the null-point; this puts a twist into the phosphor-bronze strip, and when the beam rises and clamps the upper bar, the torsion comes into play and brings the lower bar under the upper one; the beam then descends and makes contact at this point, and if any current flows through the galvanometer, there is further movement until the null-point is reached. Any alteration in the resistance of the wire under experiment causes a movement of the zero-point on the bridge-wire, and this is followed by the lower arm. The position of the lower arm can be directly indicated by means of a scale.

Prof. S. P. THOMPSON asked how the scale was calibrated.

Mr. WHETHAM said the scale was arbitrary, but it could be calibrated by the known resistance of the bridge-wire per unit length. Extension of the range can be obtained by shunting the bridge-wire with various resistances.

Mr. GLASEBROOK asked how sensitive the bridge was.

Mr. WHETHAM said that, working with a dry cell, it could easily indicate 10^{-9} on a platinum thermometer.

Mr. BLAKESLEY pointed out that if the cell was connected up the wrong way, the zero-point would be an unstable one.

A paper on "The Liquefaction of Hydrogen" was read by Dr. M. W. TRAVERS.

These experiments were undertaken in order to provide liquid hydrogen in sufficient quantity for the separation of neon from the helium with which it is usually mixed. The separation is effected by cooling the gases to the temperature of hydrogen boiling at atmospheric pressure.

The principles and conclusions do not differ from those of Dewar, but as the production of liquid hydrogen is neither difficult or costly, an account of the experiments is given.

In 1884, Wroblewski showed that strongly cooled and compressed hydrogen on being allowed to expand formed mist or spray in the tube; and, later, Olazewski repeated these experiments on a larger scale, and determined the temperature of the liquid. Other methods of liquefying hydrogen have been suggested by Lord Rayleigh and Kammerlingh Onnes. In the case of many gases a fall of temperature takes place on free expansion, but under ordinary circumstances the temperature rises in the case of hydrogen and helium. The principle of free expansion was first applied by Hampson and Linde to the liquefaction of air. Within the last two years, Dewar has shown that at a temperature close to -200°C . hydrogen behaves as an imperfect gas, and becomes cooled when allowed to expand. This principle has been applied by Dewar to the liquefaction of hydrogen in quantity. In the author's experiments, hydrogen under a pressure of 200 atmospheres passes through a coil which is cooled to -80°C . by a mixture of solid carbonic acid and alcohol. It then enters another coil contained in a chamber which is continually replenished with liquid air. The lower portion of this coil passes into another chamber, which is closed and communicates through a pipe with an exhaust pump. Liquid air flows continuously from the first chamber into the second through a pin valve controlled by a lever. The liquid air, boiling under a pressure of 100 m.m. of mercury, lowers the temperature to -200°C . The gas then passes into a regenerator coil, which is enclosed in a vacuum vessel, and, expanding at a valve, passes upwards, through the interstices of the coil and the annular space surrounding the chambers through which the gas first passes, to an outlet whence it can return to the main supply pipe. The liquid which separates from the gas is ultimately collected in a vacuum vessel.

The apparatus, with the exception of the compressor, motor, and Hampson air liquefier, is comparatively inexpensive. About £50 is required for the additional apparatus, and each time liquid hydrogen is made involves a further expenditure of about a sovereign.

Dr. HAMPSON said he would like to offer a correction. Dr. Travers had said that he (Dr. Hampson) was the first to liquefy air by the application of the counter current process to the Joule-Thomson effect. Although he was the first to make the proposal, he was not the first to apply it. He made the proposal to Prof. Dewar's assistant in 1894, and air was liquefied by Prof. Dewar by this method. Dr. Travers had referred at length to a valve which he (Dr. Hampson) had devised, but as it was straightforward common sense he did not wish to accept any credit for the use it had been to the author in his experiments. He would like to call attention to the remarkable features of the work in two respects:—The economy of means and the magnitude of results. By means of liquid hydrogen Prof. Ramsay and Dr. Travers had succeeded in obtaining the physical and other properties of some of the rarer gases.

Prof. S. P. THOMPSON said the author had asserted that the Joule-Thomson effect for hydrogen changes in sign at some temperatures, and expressed his interest in the fact that it was possible to get a cooling effect by allowing hydrogen to expand.

Mr. BOYS asked if it was necessary or desirable to allow the hydrogen to expand to atmospheric pressure.

Dr. TRAVERS said the mechanical advantages of this were great.

Dr. LEHFELDT asked if there had been any attempt to determine the temperature of the liquid, and, secondly, if the apparatus could be employed to determine the magnitude of the Joule-Thomson effect.

Dr. HARKER asked if the temperature at which the Joule-Thomson effect changes sign was known.

Dr. DONNAN said that the effect changed sign at the temperature at which " ρ " was a minimum.

Dr. TRAVERS, in reply to Dr. Lehfeldt, said he had not

determined the temperature of the liquid, and the apparatus was not suitable for measuring the Joule-Thomson effect. He should say that the change of sign occurred about -150°C . It was Daniel Berthelot who first pointed out that the change of sign corresponded with the minimum value of " β ," but the experiments of Amagat on the relation between pressure and volume were not sufficiently accurate to fix the temperature.

A paper on "*The Anomalous Dispersion of Carbon*," by Prof. R. W. WOOD, was taken as read.

Experiments were made with smoke-films, and with films deposited on plate-glass in a vacuum by an incandescent lamp. The dispersion was first measured with a Michelson interferometer, illuminated with monochromatic light of various colours obtained by prismatic analysis. The fringes were photographed and measured, readings being obtained between wave lengths 0.000040 c.m. and 0.000066 c.m. The results show a steady increase of refractive index from blue to red. The refractive index for sodium light was measured by estimating the thickness of the film and the fringe displacement, and was found to be 2.2. A prismatic deposit of smoke was then made by allowing a piece of plate-glass to move uniformly backwards and forwards over the top of a small flame. The deviation produced by this prism was measured by means of a direct vision spectroscopic, with the prisms removed. Experiments were performed with red and blue light. The mean deviation of red and blue was taken for sodium light, and this result was in good agreement with the deviation obtained by the interferometer method.

A paper on "*The Refraction of Sound by Wind*," by Dr. E. H. BARTON, was taken as read.

Assuming that the wind is everywhere horizontal, and does not vary in any one horizontal plane, but is different at different levels, then the following results are obtained for rays in the same vertical plane as the wind:—1. The direction of propagation is not usually at right angles to the wave-front where there is a wind; consequently, the cosecant law for the wave-front needs supplementing by another expression giving the direction of the ray. 2. Total reflection cannot occur if the wave-front is initially horizontal. 3. In a region where the horizontal wind increases uniformly as we ascend, the rays instead of forming a catenary describe a more complicated curve, which, however, reduces to a parabola in the special case of rays whose wave-fronts are horizontal. In the paper the relation between direction of propagation and wave-front is first worked out, and then the refraction of waves and rays on crossing into a new wind-zone is considered. This principle is then applied to the diffraction through any number of parallel wind-zones, and it is shown that the final inclinations of wave-fronts and rays are independent of the characteristic constants of the intermediate zones. It is shown that since a cosecant cannot have a value between $+1$ and -1 , total reflection becomes possible. If, however, the wave-front is initially horizontal there is no refraction of the wave-front, and no total reflection; but the ray deviates without limit from the vertical, and tends to correspond with the wave-front. When reflexion occurs it follows the ordinary optical law.

The Society then adjourned until December 14th, when the meeting will be held at the Royal College of Science, South Kensington.

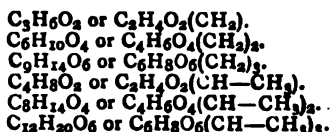
Simultaneous presence of Saccharose and Gentianose in the Fresh Root of Gentian.—E. Bourquelot and H. Hérissey.—The simultaneous presence of gentianose and saccharose in the same vegetable organism has not been found before. The author, from a study of previous experiments on gentianose, a sugar whose exact constitution has not yet been fixed, believes that the cane-sugar which is found in the fresh root of the gentian is produced by a peculiar splitting-up of the gentianose itself.—*Comptes Rendus*, cxxxi., No. 19.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 19, November 5, 1900.

Acetals of Plurivalent Alcohols.—Marcel Delépine.—The author determines the heats of combustion and formation of the glycol formals and acetals having formulae—



The formals of these three poly-alcohols, besides the acetals of glycol and of mannite, are known already, but the author prepares them by a new and easy method. The numbers obtained by him show that, in the same way as the monovalent alcohol acetals, the formation of plurivalent alcohol acetals is also a limited reaction.

Constitution of Nitrated Derivatives of Ethyl Dimethylacrylate: Ethyl Nitroacetate.—L. Bouveault and A. Wahl.—In a former paper the authors showed that fuming nitric acid transforms ethyl dimethylacrylate into a substance possessing the composition of its nitrated derivative. This compound does not dissolve in dilute cold alkalis, but alcoholic potash transforms it into the potassium salt, of a different nitrated derivative, this latter being the isomer of the first and soluble in dilute alkalis. The first the author calls α - and the second β -dimethylacrylate of ethyl. These two substances are different in that the second alone contains a negative atom of hydrogen, capable of being replaced by potassium. There actually exist two nitroacetic ethers having equal rights to the name. The authors contend that their α -nitro-methylacrylic ether, but not its β isomer, is split up by hydration into acetone and nitroacetic ether. They show that the group NO_2 is in the molecule united with carbon, and directly to the same atom of carbon which holds also the group $\text{CO}_2\text{C}_2\text{H}_5$.

A New Glucoside extracted from the Seeds of Erysimum, of the Crucifer Family.—MM. Schlagdenhauffen and Reeb.—The authors' experiments show that the seeds of *Erysimum aureum*, an ornamental garden plant, contain two active principles—one of an alkaloidal nature which provokes paralysis, and the other a glucoside, which constitutes a violent poison, affecting the heart.

MISCELLANEOUS.

The Vapour-density of Sulphur.—O. Bleier and L. Kohn.—The authors have measured the vapour-density of sulphur at reduced pressure, and arrive at the conclusion that the lower the temperature the more the condensation of the molecule of sulphur approaches S_8 , as shown by the following table:—

Temperature.	Pressure. M.m.	Density of the vapour with regard to oxygen.
Boiling-point of—		
Diphenylamine, 310° ..	42.6	7.44
Benzoate of amyl, 262° ..	15.0	7.50
Quinolein, 236° ..	9.4	7.66
Benzoate of ethyl, 212° ..	4.2	7.80
Dimethylaniline, 193° ..	2.1	7.85

—*Berichte*, xxxiii., p. 50.

Society of Arts.—The Juvenile Lectures at the Society of Arts will be given this Session on the afternoons of Wednesday, January 2 and 9, by Mr. E. Walter Maunder, F.R.A.S., Superintendent of the Solar Department, Greenwich Observatory. The subject of the course will be "Eclipses."

MEETINGS FOR THE WEEK.

MONDAY, Dec. 3rd.—Royal Institution, 5. General Monthly Meeting.
TUESDAY, 4th.—Society of Arts, 8. (Cantor Lectures). "Electric Oscillations and Electric Waves," by Prof. John A. Fleming, M.A., D.Sc., F.R.S.

WEDNESDAY, 5th.—Society of Arts, 8. "Road Traction," by Prof. H. S. Hele-Shaw, LL.D., F.R.S.

THURSDAY, 6th.—Chemical, 8. Ballot for the election of Fellows.
"Santalonic Acid," by Alfred C. Chapman.
"Ammonium Bromide and the Atomic Weight of Nitrogen," by A. Scott, F.R.S. "Interaction between Urethanes and Primary Benzenoid Amines," by A. E. Dixon, M.D. "The Decomposition of Chlorates—Part III., Calcium Chlorate and Silver Chlorate," by W. H. Sodas, B.Sc. "Nitride of Iron," by Gilbert J. Fowler, M.Sc. "The Heat of Formation and Constitution of Iron Nitride," by Gilbert J. Fowler, M.Sc., and Philip J. Hartog, B.Sc. "Relationships of Oxalacetic Acid," by H. J. H. Fenton, F.R.S., and H. O. Jones, B.A., B.Sc.

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MICHAELMAS TERM.—Monday, October 1, to Saturday December 15.

LENT TERM.—Monday, January 7, to Saturday, March 30.

EASTER TERM.—Monday, April 28, to Saturday, July 27.

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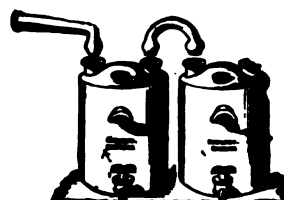
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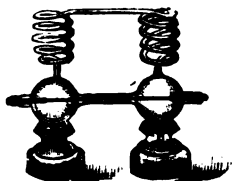
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THE CHEMICAL NEWS.

VOL. LXXXII., No. 2141.

THE ANALYSIS OF FERRO-SILICON AND SILICO-SPIEGEL.

By FRED. IBBOTSON and HARRY BREARLEY.

Total Carbon.

THE powdered alloys are hardly at all attacked by cold, and only partially by hot copper solutions, but they are easily and completely decarbonised by ignition in a stream of oxygen, either with or without such oxidising reagents as copper oxide and lead chromate. Better without, because there is then no reagent blank to be determined and allowed for, and there are no metallic beads (Cu or Pb) formed which may occlude some of the unattacked sample. As there is generally no fusion, the same boat is available time after time. The ferro-silicon after ignition is powdery or easily crumbled; silico-spiegel is fritted into a single cake.

Graphite.

The carbon in ferro-silicon exists chiefly as graphite, the combined carbon rarely amounting to more than a few tenths per cent.

	Lonsdale.	Ayresome.	Mostyn.
Combined C	0.20	0.15	0.19
Graphite	1.39	1.39	1.62
Silicon	12.32	13.28	12.08

The graphite is determined as follows, combined carbon being obtained by difference:—Cover 2 or 3 grms. of the powdered alloy with 70 to 100 c.c. of nitric acid (1.20), and heat nearly to boiling; the reaction with ferro-silicon is very slight. Add a few drops of hydrofluoric acid; the reaction becomes very vigorous. Keep up the reaction by adding small amounts of hydrofluoric acid until only particles of graphite are to be seen. Do not hurry the reaction; give the flocks of the nitrated (combined) carbon compound time to dissolve in the nitric acid before any considerable excess of hydrofluoric acid is added. The sample may be dissolved in glass vessels without injuring them; they are preferable to platinum ware, in that they permit the course of the reaction and the complete decomposition to be readily observed. Filter the graphite on to asbestos, wash first with water, and then with boiling soda hydrate in order to dissolve out any carbonaceous matter not graphite, wash once with dilute hydrochloric acid, and finally with water until the washings are free from chlorine. Burn in a current of oxygen, in the usual way.

Donath and Hailsig (*Stahl und Eisen*, xvii., 670, and *J. I. S. I.*, 1897, ii., 488) examined a ferro-silicon which contained 14.3 per cent silicon and as much as 1.39 per cent combined carbon, which the authors believe was present in the alloy as carbide of silicon (carborundum). In the process given below for estimating silicon, carborundum, if present, would be with the silica, and after treatment with hydrofluoric acid would still be in the residue undecomposed. This residue, however, in our experience, never exceeds 2 or 3 m.grms., and consists chiefly of ferric oxide; so that generally the existence of carbide of silicon in ferro-silicon, for other reasons as well as that just given, is very unlikely.

■ The total carbon of silico-spiegel is generally higher than that of ferro-silicon, and contains more combined carbon. The graphite and combined carbon are estimated as above. Some examples are:—

	I.	II.	III.	IV.
Combined C. ..	1.89	1.61	1.31	0.51
Graphite	0.34	0.94	0.60	1.22
Silicon	10.53	10.95	12.10	13.34
Manganese ..	19.85	18.14	18.25	20.20

Silicon.

The statement that these alloys cannot be completely decomposed by any mixture of the acids, but must be attacked by fusion with KHSO_4 , Na_2CO_3 , or Na_2O_2 , is erroneous. They are very readily attacked by a mixture of nitric and hydrochloric acids.

Hogg (*CHEM. NEWS*, lxvii., 27) points out that, after evaporation of the acid liquid and treatment of the residue with hydrochloric acid, the silica is always discoloured, not because the alloy has been incompletely decomposed, but because the physical condition of the silica is so peculiar that when heated with an iron salt, in the dry state, it takes up and firmly retains some ferric oxide. Hogg also shows that for alloys containing from 10 to 15 per cent of silicon the amount of Si which goes into solution lies between 0.1 and 0.3 per cent: this amount he omits to recover, and adds, by way of correction, 0.2 per cent to that obtained by filtering as soon as the decomposition is completed. A large number of test analyses enable us to confirm Hogg's conclusions in every particular, save the minor one referred to in the following account of the process:—

Weigh off 2 grms. of the finely-powdered alloy, add 50 c.c. concentrated HCl and 10 to 20 c.c. HNO_3 ; boil to decomposition, which needs 10 to 15 minutes; add about twice the volume of water and filter at once, wash with dilute HCl, ignite, and weigh. To the result obtained add only 0.1 per cent as a correction for the soluble silica.

The amount of silicon which goes into solution is increased by boiling the solution after diluting.* By allowing the diluted solution to stand on the hot plate for two hours, between 20 and 30 m.grms. SiO_2 were dissolved: the correction, therefore, of 0.1 per cent applies only when the filtration is proceeded with immediately after diluting. The silica as weighed is in a very fine state of division, and is also very hygroscopic.

Manganese.

The alloys are most readily decomposed by nitrofluoric acid. In ferro-silicon the manganese is often so low that it can be estimated by oxidising with red-lead and titrating the permanganic acid with FeSO_4 , &c. Not more than 0.01 grm. Mn can, however, be assayed by this process.

Reddrop and Ramage (*Journ. Chem. Soc.*, lxvii., 268) use soda bismuthate in cold solutions, in order to convert manganous oxide to permanganate. This process works as well for large as for small amounts of manganese:—Dissolve 1 grm. of the alloy in 30 c.c. 1.20 HNO_3 , and 1 or 2 c.c. HF; make quite cold, add 10 c.c. water, and then add about 2 grms. of soda bismuthate. Filter through asbestos, add standard H_2O_2 , and titrate with N/10 permanganate. For silico-spiegels dilute the dissolved alloy to 100 c.c., measure out 25 c.c., add 25 c.c. 1.20 HNO_3 , oxidise as before, filter into H_2O_2 ,† and titrate the excess with KMnO_4 . For a full description of the process the original paper must be referred to, the present object being merely to note that silicon alloys are most conveniently prepared for the estimation of the manganese by decomposing with fluor-nitric acid, and that the use of hydrofluoric acid does not interfere with any phase of the bismuthate process. Needless large amounts of HF, however, may cause the final permanganate tint to be readily converted, through the agency of manganous fluoride, into manganic oxide.

* A. H. Allen has shown that when irons are attacked with HCl or H_2SO_4 , part of the silicon is liberated as "leucosil," which is soluble in dilute acids.

† Reddrop and Ramage offer no reason why, for steels and low manganiferous alloys, the hydrogen peroxide is added to the permanganate and for spiegele and highly manganiferous alloys the permanganate is filtered into the peroxide. The distinction is a necessary one; can it be explained?

Phosphorus.

We have described a process for estimating phosphorus (CHEM. NEWS, lxxii., 55) depending on the conversion of the Mo in the $\text{Am}_2\text{PbO}_4 \cdot 12\text{MoO}_3$ into PbMoO_4 , but it cannot be straightaway applied to these alloys, because neither of them are decomposed by 1.20 nitric acid.

By decomposing with nitro-hydrochloric acid, and evaporating so as to dehydrate the silica and eliminate HCl, a portion of the P always remains with the SiO_2 . This may be a source of very serious error when highly phosphoretted material is being assayed. As an example we may quote a silico-spiegel, where of the 0.20 per cent P present 0.063 per cent was found with the ignited silica. An obvious means of minimising, if not altogether eliminating this error, is to filter as soon as the sample is decomposed, as in the estimation of silicon, and use the evaporated filtrate for the estimation of phosphorus.

A more expeditious way of estimating the phosphorus is suggested by the easy decomposition of these silicon alloys with nitrofluoric acid. To decompose the silica in the solution would at once allay all fear of P being filtered off. But the precipitation of P as ammonium phospho-molybdate is by no means insensible to the presence of hydrofluoric acid. A number of experiments made with dilute solutions of soda phosphate showed that small amounts of HF retarded the precipitation of the phospho-molybdate, and larger amounts altogether prevented it. Much the same thing happens when the nitric acid solution of a steel from which P is to be precipitated is treated with hydrofluoric acid.

By preparing identical solutions of the same steel which contained 0.064 per cent of phosphorus, and adding 17.34, and 51 c.c. hydrofluoric acid just before adding the Am_2MoO_4 reagent, we found the first to be precipitated at once, but the second and third to show no signs of a precipitate for some time. After standing for two hours at about 60° the filtered precipitates were converted to PbMoO_4 , and found to correspond to 0.060, 0.063, and 0.061 per cent phosphorus.

Along with the usual amount of nitric acid, a ferro-silicon or silico-spiegel can be easily decomposed by using 4 c.c., or less, of hydrofluoric acid; what portion of this is unused by the silicon of the alloy is largely eliminated by the subsequent boilings, so that a series of tests made by adding pure silica to the steel being dissolved by nitrofluoric showed no signs, when the phosphorus came to be precipitated, that any hydrofluoric acid had been used, and, on completing the estimations, gave 0.065, 0.063, 0.064, and 0.066, instead of the 0.064 per cent P actually present. It is clear then that hydrofluoric acid may be safely used. The process is most conveniently carried out as follows:—

To 2 grms. of the powdered alloy add 45 c.c. 1.20 nitric acid, and 25 to 30 drops hydrofluoric acid. When, without the application of heat, the reaction subsides, add another 25 to 30 drops HF. What little of the alloy is then decomposed goes on boiling. After complete decomposition add permanganate until a permanent MnO_2 precipitate is formed, clear with FeSO_4 , filter off the graphite, add 6 or 7 c.c. strong ammonia, precipitate with Am_2MoO_4 reagent, and weigh finally as PbMoO_4 as described in our previous paper.

Hydrofluoric acid may contain small amounts of phosphoric acid. It is estimated (and allowed for) by evaporating 100 or 200 drops of the acid with sulphuric acid in a platinum dish until SO_3 fumes are given off, transferring to a glass vessel, oxidising with permanganate, clearing with FeSO_4 , adding AmHO to precipitation of Fe, re-dissolving in HNO_3 , precipitating with Am_2MoO_4 mixture, and finishing as usual.

We have never found ten times as much permanganate and ferrous sulphate as are used for each assay to contain an appreciable amount of phosphorus.

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SCHÖFFEL'S PROCESS FOR ESTIMATING TUNGSTEN IN STEEL.

By ERNEST BAGLEY and HARRY BREARLEY.

SCHÖFFEL's process for estimating tungsten in steel does not meet with anything like the practical appreciation it deserves if we may take the processes commonly described in text-books as indicating the practice of works' laboratories.

The process (CHEMICAL NEWS, xli., 31) depends on the fact that a neutral solution of a double copper salt, such as is often used for liberating carbon from iron, leaves all the tungsten unattacked. After ignition of the carbonaceous residue and removal of the silica, the solution of the fused tungstic oxide is precipitated with mercurous nitrate.

Besides carbon, tungsten, and silicon, the residue will also contain sulphur, associated with iron and manganese; chromium, together with considerable iron; titanium, when it happens to be present in the steel; and, if the copper solution is quite neutral, all the phosphorus.

Sulphur and phosphorus are usually present in such small amounts that no appreciable error could be introduced by volatilising the silica with hydrofluoric acid, fusing the residue with soda carbonate, filtering off and weighing the small amount of iron or manganese, and arriving at the percentage of tungsten by difference. Such a procedure gives quite accurate results when applied to steels in which tungsten only is the uncommon element. But in case chromium were present, the iron to be filtered off after the soda carbonate fusion would be a large amount, and the chromium would pass, partly at least, into solution. Under such unfavourable circumstances, if the operations are carefully performed, very tolerable results are obtainable by titrating the chromic acid in the acidified filtrate, adding its Cr_2O_3 equivalent to the mixture of $\text{Fe}_2\text{O}_3 + \text{Cr}_2\text{O}_3$ which has been filtered off and ignited, and deducting the total from the weight before fusion, so as to arrive at the amount of tungstic oxide.

It may occasionally or even frequently happen that the tungstic oxide found by difference in the above manner is less than the amount of impurities associated with it; and in this fact, at a time when chromium and tungsten are so often associated in steels, lies the weakness of Schöffel's process for general purposes.

By decomposing chrome-tungsten steels with cupric solutions which are made increasingly acid with HCl, the amount of impurity to be found in the ignited carbonaceous residue becomes less and less. By using solutions which contained one-third their volume of strong hydrochloric acid, we have obtained such residues as contain no very objectionable amount of either iron or chromium and all the tungsten.

Weigh off 5 grms. of the sample, add 50 grms. cuprammonium chloride crystals, 100 c.c. hot water, and 50 c.c. strong hydrochloric acid. By digesting at boiling-point, with occasional shaking, the precipitated copper dissolves in half an hour or so, and after standing a little longer, the solution is filtered through paper pulp. Wash with dilute HCl, ignite, volatilise silica with HF, fuse with Na_2CO_3 , dissolve in water, and ignite the separated Fe_2O_3 , &c. The filtrate may have a faint yellow colour; if so, acidify with H_2SO_4 , add FeSO_4 , titrate with KMnO_4 , calculate to Cr_2O_3 , and deduct along with the Fe_2O_3 in order to arrive at the percentage of tungsten.

That all the tungsten remains with the residue when strongly acid copper solutions are used, is shown by the following analyses:—

Percentage tungsten by—

Evaporating with acids or other standard process.	Digesting with 33 per cent HCl cuprammonium chloride.
4.50	4.52
10.06	10.09
3.41	3.44
1.62	1.62

And the amount of impurity ($\text{Fe}_2\text{O}_3 + \text{Cr}_2\text{O}_3$), of which account has to be taken, may be seen from the following:—

Percentage tungsten.		Percentage Cr present.	Weight in the residue of—	
Standard process.	HCl.Cu.		Cr_2O_3 .	Fe_2O_3 .
8·81	8·81	2·25	0·0020	0·0100
6·28	6·33	0·80	trace	0·0060
—	3·32	2·51	0·0008	0·0050
3·43	3·40	1·44	trace	0·0050

When steels which contain both tungsten and molybdenum are treated in this way, the molybdenum may go wholly into solution on continued digestion, but this is not invariably the case.

When the tungsten is but a fraction of 1 per cent, the treatment at boiling-point with solutions containing one-third their volume of strong hydrochloric acid causes the results to be slightly low. In such cases treatment of the steel with cuprammonium solutions containing only 10 per cent of strong hydrochloric acid is preferable.

Part of the silicon passes into solution, and cannot therefore be estimated by measuring the loss in weight on treating with hydrofluoric acid.

SOME RESULTS OBTAINED IN THE ELECTROLYSIS OF FUSED SALTS.

By F. MAXWELL LYTE.

A PATENT of the writer's for the electrolysis of fused lead chloride was being experimented on by Mr. O. J. Steinhart (Chemist to the Chemical and Electrolytic Syndicate), which company had taken over several of the writer's patents with the object of working them. These experiments were made on about 5 or 6 lbs. of lead chloride at a time.

The lead chloride, after being well dried, was fused in an earthen pot, into which another rather smaller, but similar vessel, was inverted, forming a sort of bell with its lower part dipping into the molten chloride. Two carbon rods intended to serve as cathode and anode were cemented gas-tight into the top of the bell, with a cement composed of sodium silicate and china clay, with their lower ends dipping an inch or two into the fused lead chloride. A peep-hole was also arranged in the top of the bell so as to be able to look down into it and see what passed.

The writer himself was unable to be present owing to illness, but his son, Mr. C. Maxwell Lyte, though neither a chemist nor an electrician, was able to report all that occurred with so much clearness and exactitude, that the writer almost felt as if he himself had assisted at the operations.

On passing the electric current at a pressure sufficient to cause the electrolysis of the fused lead chloride, a strange phenomenon presented itself. The space between the electrodes at once became occupied by what these gentlemen characterised as a kind of smoke or cloud, emanating apparently from the immediate neighbourhood of the cathode, and growing less and less intense as it approached the anode, in the vicinity of which it apparently disappeared altogether.

During the whole experiment little or no chlorine was obtained, only a small amount of a colourless and odourless gas, which later on was found to be oxygen, being evolved from the anode, while from time to time a little metallic lead was seen to drip from the cathode.

All or most of this lead was probably derived from the decomposition of the lead oxide in the lead oxychloride, formed in the fused lead chloride, which dissolves it, by the absorption of atmospheric oxygen. Lead and oxygen have a lower heat of combination than lead and chlorine, and consequently the lead oxide would decompose first,

Casting about in order to find some means of preventing this reaction, and of avoiding the re-combination of the lead with the chlorine which evidently occurred, and which obviously would result in a considerable loss, the writer came to the conclusion, which seemed almost self-evident, that if instead of using a carbon cathode, the fused metal itself were made the cathode, the lead resulting from the electrolysis would be no longer diffused into the electrolyte, but would be deposited on and amalgamated with the fused lead cathode, and that thus the so-called cloud or smoke no longer being formed, that cause of loss would disappear.

This we at once tried, and it resulted apparently in a complete success, but when afterwards the experiment was tried quantitatively, it was found that there was still a loss of about 10 per cent of the chlorine that should theoretically be produced. This may have arisen from an absorption of oxygen, as already mentioned, or from leakage, but nevertheless it would be found difficult in practice to avoid all loss. The matter formed the subject of two successive patents by the writer, one for the electrolysis of fused lead chloride with a carbon anode and a fused lead cathode, No. 7264, April 8, 1893, and later on another for the electrolysis of fused zinc chloride with the same arrangement of the electrodes, No. 15,813, August 22, 1895.

The lead chloride patent was ultimately abandoned for various reasons, notably, among others, from the difficulty of obtaining the lead chloride on the large scale with sufficient ease and economy.

Zinc chloride, on the other hand, can be easily produced from the ore or from the zinc oxide resulting from its treatment, and the zinc chloride patent offers besides this several other advantages.

The process has lately undergone a very long and exhausting trial on a large scale, and is soon now about to be worked for manufacturing purposes.

The above-mentioned smoke or cloud of metallic lead, forming between the electrodes and disappearing as it approached the carbon anode, affords a ready explanation of the insurmountable difficulties which seem to attend all attempts of the electrolysis of aqueous solutions of the salts of the more readily oxidisable metals, such as potassium, sodium, zinc, &c., where a solid cathode is employed, zinc when in the nascent or finely divided state decomposing water with peculiar facility.

The analysis of the black deposit resulting from the electrolysis of aqueous solutions of zinc salts has shown it to be a mixture of metallic zinc and zinc oxide, just such as we might expect to find with a metal the heat units evolved by which in its combination with oxygen so nearly corresponded with those evolved by hydrogen in like circumstances.

The writer would not now allude to the above-mentioned experiments, which date from some seven or eight years back, and which were thought hardly worth publication at the time, they not seeming in themselves to have sufficient scientific interest, and he would simply have contented himself with having obtained the two patents above mentioned, had not the subject again cropped up lately in the experiments of A. Helfenstein, related in the number for July, 1900, of the *Journ. Chem. Soc.* (Abstracts, Part II., p. 383). He seems to have operated in an ordinary V-tube, and not on nearly so large a scale as we did, using a fused lead cathode. Consequently, he did not get the cloud of metallic lead which we did when working with a carbon cathode.

In Faraday's account of his own experiments respecting the electrolysis of fused lead chloride, if the writer's memory is correct, that great philosopher also remarked that the amount of chlorine evolved did not correspond theoretically with the amount of current as indicated by the voltmeter, and it seems just as likely, if the report in the Society's Journal is correct, that the extra amount of current consumed should be attributable to the electrolysis of the lead oxide in the lead oxychloride above mentioned,

as to the solution of the metallic lead in the fused lead chloride.

Indeed, this latter seems to the writer most improbable. The presence of the cloud of metallic lead in the electrolyte under the circumstances above stated, and the evolution of oxygen (not of chlorine) at the anode, while a little lead dripped from the carbon cathode, all seem to negative such a conclusion, and point to a far simpler way of accounting for the apparent anomaly than by finding in it an exception to Faraday's law.

ON NATIVE TELLURIUM FROM THE HANNAN'S DISTRICT, WESTERN AUSTRALIA.

By R. W. EMERSON MACIVOR, F.I.C., &c.

WHILE examining some specimens of telluride ore from the Hannan's district, Western Australia, I found, associated with pyrites, a finely granular, white, metallic-looking mineral, which, on analysis, proved to be tellurium. Freed from foreign substances it had a specific gravity of 6.2 and contained—

Te..	..	..	..	..	96.935
Au..	..	..	..	..	2.399

99.334

Iron and sulphur were present, but I failed to detect any trace of selenium.

In only one other locality in Australia has native tellurium been found, viz., in the Ballarat district, Victoria.

The Chromium Syndicate, Ltd.,
Quality Court, Chancery Lane, W.C.

ON CHLORINE HEPTOXIDE.

By ARTHUR MICHAEL and WALLACE T. CONN.

THE marked increase in stability shown by non-metallic oxides and acids with increment of oxygen led us to re-investigate perchloric acid, with a view of preparing its anhydride. For this purpose a pure acid is necessary, and as the product prepared according to the directions given by Roscoe (*Journ. Chem. Soc.*, xvi., 82), is somewhat impure, and can be obtained in this way only with a great loss of material, we modified the method by heating the perchlorate and sulphuric acid in a vacuum. Portions of 25 grms. of salt and 100 grms. of sulphuric acid (of about 1.839 specific gravity at 15° C.) were brought into a fractionating flask,* whose low lateral tube is connected with a second flask placed in a freezing mixture, and heated under 10–20 m.m. pressure in a paraffin bath. The reaction starts at about 90° C., and perchloric acid begins to pass over when bubbles are noticeable. The heating should be gradual to prevent frothing. In about an hour the bath may be raised to 160°, and it is kept at that temperature until all the perchlorate is dissolved, the operation usually taking about two hours. It was found impossible to decompose all the perchlorate, as the process becomes reversible towards the end, and 1.5–2 grms. of salt are easily regained from the cooled contents of the flask. The crude acid contained traces of sulphuric and hydrated perchloric acids, which were removed by a subsequent fractionation of the freshly prepared substance in a vacuum. The yield was 85–90 per cent of the theory, if allowance was made for the regained salt.

The acid was usually very slightly coloured and differed in some of its properties from the substance described by

Roscoe. Under 11 m.m. pressure its vapour heated a thermometer to 19° C. In a glass-stoppered bottle the colourless oil coloured on standing, even when kept from light, and after three weeks was quite dark, but this sample did not explode, although it was kept several months. In contact with paper or wood it exploded with a slight blue flame, carbonising but not igniting the material. In small amounts it could be mixed with well-cooled absolute alcohol without explosion, apparently with ester-formation, as was also the case when dry ether was used. To 5 grms. dry benzene, placed in a freezing-mixture, 1 grm. acid was added, and the cooled tube sealed. The acid dissolved, forming a green solution, which soon deposited a carbonaceous substance, increasing in amount until the green colour disappeared. The tube opened under slight pressure, and no free acid could be detected in the solution. Iodine dissolved in the well-cooled acid, using the proportion of 0.5 atom to 1 molecule, to form a dark solution, which, exposed to bright light, gradually changed into an almost white substance. The tube opened under slight pressure, and the gas contained a little chlorine. On heating the substance gave off iodine, leaving a white body, which, after dissolving in water, showed tests for iodic acid.*

The most interesting behaviour of perchloric acid is towards phosphorus pentoxide, and to perform this experiment successfully it is necessary to adhere strictly to the following directions:—In a small glass-stoppered retort, connected with a drying-tube filled with phosphorus pentoxide and placed in a freezing-mixture of ice and salt, 10 grms. of phosphorus pentoxide are brought, and, by means of a long tube which is bent inward and contracted at the delivering end, and has a rubber ball attached to the other end, not more than 10 drops of perchloric acid are added, waiting ten minutes before adding a second portion. It is advisable to allow the acid to drop on the sides of the retort, and the temperature of the freezing-mixture should be kept below –10° C. The retort, left in the freezing-mixture, is allowed to stand for a day, then connected with a well-cooled receiver, and slowly warmed in a water-bath to 85° C., when the new oxide passes over. The product obtained in this way is practically pure, and may be used for the experiments described below, but the freshly prepared substance may be re-distilled under ordinary pressure without danger, when it passes over at 82° C. (corr.). It should be well borne in mind that, even though the above directions for preparing the crude oxide are followed, the apparatus may be virtually pulverised by a violent explosion, and that the personal precautions necessary for work of this kind must be always observed.

Chlorine heptoxide is a colourless, very volatile oil, that on standing a day begins to turn yellow, after two or three days is greenish-yellow, with the liberation of a greenish gas. In comparison with the previously known oxides of chlorine it shows a remarkable stability, and, although when brought in contact with a flame, or by a sharp percussion, it explodes with great violence, it may be poured on paper, wood, or similar organic matter with impunity, the oxide simply volatilising in the air. Brought into a well-cooled tube with some stick sulphur and the tube corked, no reaction occurred, even after standing several days; and what is a more striking evidence of its stability, it may be poured on a cooled piece of phosphorus and remain for some days without being attacked. With cold water it sinks to the bottom of the vessel and passes slowly over into perchloric acid, but in a closed vessel it requires some days' standing before the peculiar odour of the oxide has disappeared. Dry and cooled benzene dissolves the oxide, and after a short time a reaction ensues. With iodine a reaction occurs slowly in the dark, more rapidly in the light, with liberation of chlorine and

* The neck of the flask was contracted and the air-tube fitted in tightly by means of asbestos paper, which was covered by a piece of rubber tubing; a similar joint was used between this flask and the receiver. Attached to the air-tube was a P_2O_5 drying-tube, and between the receiver and manometer a calcium soda lime tube.

* In the last number of the *Annales* (ccc., 369), which we have just received, Vorländer and von Schilling describe a similar method of preparing the acid, although their yield is not as favourable as ours. Their acid also appears to be somewhat more explosive, which may be due to their using a perchlorate not entirely free from chlorate.

formation of a white solid. This substance begins to decompose at $380^{\circ}\text{C}.$, forming iodine and oxygen, and apparently represents the heptoxide of iodine. Bromine, under similar conditions, is without action.*—*American Chemical Journal*, xxiii., No. 5.

THE ALLOTROPIC FORMS OF SELENIUM.†

By A. P. SAUNDERS.

(Continued from p. 264).

Conclusions from Dilatometric Experiments.

It may seem that the experiments with the dilatometer have been needlessly multiplied. This repetition is due in part to the fact that the method of working was at first very imperfect, but mainly to the fact that the evidence to be obtained from them is both positive and negative. There is to be found in many of the text-books a description of the gray form of selenium obtained by heating the vitreous modification to 200° , which separates this as a distinct modification from the black crystals obtained in other ways, and these experiments were begun with the expectation of finding two such distinct forms with a real transition point. It will be seen, however, that the evidence is all against the existence of any stable form except the one having a density of 4.8 and the multiplicity of the experiments only serves to render that evidence more conclusive. When selenium is heated in liquids in which it is somewhat soluble, like quinoline or aniline, or in which it is practically insoluble, as paraffin, only two modifications are obtained, the vitreous and the metallic. The metallic form is a stable throughout its range, up to 220° or nearly; whereas the vitreous form goes over at any temperature above about 60° to 80° according to the liquid present. When vitreous selenium is placed in water, it behaves in the same way as it does in other liquids which do not dissolve it. Furthermore, with regard to the behaviour of the element at 200° , only one form can permanently exist there. It makes no difference whether fused selenium be cooled to 200° , or whether vitreous selenium be rapidly heated to 200° , or again, whether it be allowed to go over at any lower temperature and then raised to that point—the final density at 200° is always the same, and lies along the density curve for metallic selenium. Sunlight causes no change of volume in metallic selenium either at ordinary temperatures or at higher ones, up to 200° . It is not as a rule possible to raise the temperature of vitreous selenium above about 140° without a sudden change into the metallic form, but it may occasionally, by rapid heating, be brought to 180° or above before the change takes place. When metallic selenium is kept at ordinary temperatures or placed in a bath heated to 40° , 50° , or 60° , or any higher temperature, below 220° , it shows no tendency to go over into any other form.

The general character of the curves obtained by plotting the results of dilatometric determinations with selenium is illustrated by the accompanying drawing (Fig. 2).

The line A represents the metallic form; the line B, the fusion, with formation of liquid selenium; this when cooled gives the line C; if the cooling be stopped at 200° we obtain the line C_1 leading back to A; if it be stopped at 100° , we get the line C_2 also leading back to A. Finally, if the line C be followed down to ordinary temperatures, it is not usually possible to reproduce it with rising temperature, but the change follows a curve of the general

form of D, which again leads us back to the line A, for the metallic modification.

Two objections may be urged against conclusions drawn from dilatometric measurements regarding the possible transformation of a substance. In the first place, the results only apply to cases where the material studied is in contact with a liquid, in which it will always be more or less soluble; and in the second place, the results, being really only density determinations, will fail to bring out such changes as involve very slight alteration in specific gravity. This latter objection must be let stand, although the probability is always in favour of two different modifications having differences in density which lie within the range of experimental proof. To meet the first objection, a few density determinations were made, to fill out the gaps left in earlier investigations along this line. They will be found recorded under the corresponding heading.

Vitreous selenium is stable, by itself, at ordinary temperatures, for an indefinite time. Hittorf states that it remains for years without alteration, and the fact that the selenium of commerce is in the vitreous form further demonstrates its permanence.

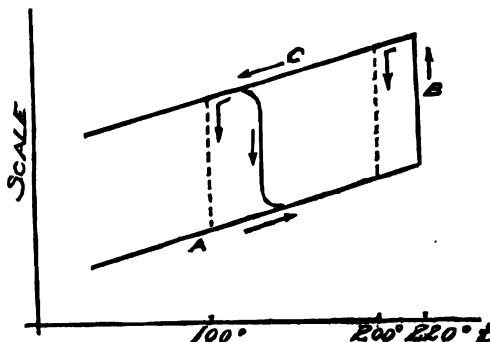


FIG. 2.—The vertical dotted lines, beginning at the right, should be lettered C_1 and C_2 . The intermediate curved line is the line D.

To illustrate the degree of its viscosity, a rod of it, having a diameter of about 5 m.m., was fastened in a horizontal position, and a weight of 200 gr. hung upon it at a distance of 6–8 c.m. from the point of support. No change could be observed in it at the end of a week. On the other hand, when the substance is fused and drawn into stout threads, and these are fastened horizontally, they bend almost at once by their own weight alone.

The vitreous and amorphous forms being both of them liquid selenium, but in different states of division, it follows that their solubilities must also be practically identical. Nevertheless, while the solubility of the amorphous form in carbon disulphide is always admitted, it will be found stated here and there that the vitreous form is nearly or quite insoluble in that liquid.

Thus Schützenberger (*Chimie Générale*, i, 438), after contrasting the solubility of the amorphous form with the insolubility of the metallic, adds:—"Vitreous selenium is also almost insoluble in carbon disulphide." But many points in the description of selenium, in this, as well as in most of the other text-books, are quite at variance with the truth, often, indeed, in disagreement with facts which have long been known. To show that vitreous selenium does dissolve in carbon disulphide, it is only necessary to place a piece in a little of this liquid for a few moments, and follow the immediate change of colour.

The data regarding the solubility in this liquid—and they apply equally for the amorphous modification—are as follows.—Mitscherlich (1855) states as a result of his experiments that one hundred parts of carbon disulphide at 46.6° dissolve 0.1 part of amorphous selenium.

* It is proposed to examine the behaviour of concentrated solutions of chloric and bromic acid towards P_2O_5 . The stability of chlorine heptoxide may be explained on lines similar to those which I (*Yourn. Prakt. Chem.*, [3], ix., 328), have given for carbon tetrachloride. This subject, which has a very important bearing on chemical affinity and on valency, will be more fully discussed in a later paper.

† From the *Journal of Physical Chemistry*, vol. iv., No. 6.

and at 0°, 0.016 part. Rammelsberg (1874) says that one part of selenium (probably amorphous) is soluble in 1376—2464—3746 parts of carbon disulphide at 20°. We have no details of either of these determinations, but Mitscherlich had noted for the vitreous form as well as the amorphous, that on standing in contact with carbon disulphide, red crystals appear, and he had interpreted this in the case of the vitreous form as not being a change in the mass of the material, but a superficial alteration involving successive solution and re-crystallisation.

To determine the correctness of this explanation I placed some pieces of vitreous selenium in carbon disulphide, and left them in diffused daylight. On the following day the surfaces were found to have lost their black vitreous lustre, and to have acquired a dull brownish iridescence; they were, moreover, everywhere covered with minute, but well-formed, glittering, transparent red crystals, some of which were loose in the bottom of the vessel, but most of them adhering to the mass of the selenium. After twelve days more, there was no further visible change. The fragments were then removed from the solution, and when broken were found to have their original black fracture within. This shows that Mitscherlich's explanation is correct. The change does not extend within, but stops as soon as the entire surface has become coated with crystals or a crystalline film. It shows also that in all likelihood the crystals are much less soluble than the original material; otherwise this change would be difficult to explain. My experiments have further shown that the change goes on much more rapidly than Mitscherlich supposed.

Now the observation just recorded goes far to clear up the discordant solubility results of Rammelsberg. It is probable that they were obtained by successive determinations from the same material, and that their variation corresponds to the change going on in the solution as it passed from a state of saturation with respect to amorphous selenium, to a state of saturation with respect to the red crystals themselves. Hence, while his first value according, as it does, fairly well with Mitscherlich's, probably represents the solubility of amorphous selenium, the second represents nothing definite at all, and the third is possibly somewhere near the value for the red crystals.

A further experiment of mine showed that vitreous selenium in carbon disulphide in the dark, remained unchanged for six days. At the end of that time, it was allowed to stand in the light for a day, and was then found covered with crystals.

Vitreous and amorphous selenium are known to be soluble in various other liquids, but few quantitative measurements have been made.

Retgers (1893), using the powder of commercial vitreous selenium, found its solubility in methylene iodide at 12°, to be 1.3 parts in 100; from this solution, on evaporation, the selenium separates in monoclinic, blood-red leaflets, and also in black opaque aggregations, the latter probably metallic selenium. Schneider (1866) found that when amorphous selenium is allowed to stand in contact with selenious bromide for eight days, with occasional heating to 70°—80°, a solution is obtained which contains in 2 gr. of the solvent—probably at ordinary temperatures—0.44 gr. selenium.

Rathke (1869), in endeavouring to find a liquid which would yield good crystals of the metallic form, noted the solubility of vitreous selenium in carbon di-selenide, selenium di-ethyl, sulphur di-ethyl, and in selenious chloride, which last is capable of taking up very considerable quantities of the element. All forms of selenium dissolve in sulphuric acid, forming a green solution, but no exact study has been made of the degree of solubility. Some further notes on solubility will be found later on, in the description of the other modifications.

Petersen (1891) found it impossible to get vitreous selenium quite pure. He found that samples prepared by rapid cooling of the fused mass were never free from material which was insoluble in carbon disulphide, though

his difficulty may possibly have arisen from impurities in the material.

Regnault, in studying the rate of cooling of melted selenium to find whether there were any irregularities in the change of temperature, made a series of observations between 260° and 40°, and found no signs of any irregularity. The apparatus consisted merely of a vessel filled with a large quantity of selenium, with a thermometer standing in the mass. In order to confirm this result, he placed the apparatus in an air-bath at 100°. The readings, which were taken every minute, are as follows:—

241.6°	175.95°	148.1°	137.85°
229.8	170.9	146.3	137.0
219.3	166.4	144.7	136.25
210.2	162.4	143.25	135.5
201.7	158.8	142.0	134.7
194.4	155.6	140.75	133.9
187.6	152.9	139.65	133.2
181.6	150.4	138.75	

From here on, the readings were made every five minutes—

133.2°	115.0°	114.05°	116.0°
129.2	113.4	115.9	113.35
125.35	112.8	117.9	111.25
121.9	112.6	120.75	109.65
119.1	112.55	121.3	108.75
116.8	113.0	118.9	107.55
			106.8

It will be seen that at about 113° there is a marked rise of temperature, followed by a uniform drop like that which preceded. What the cause of this may have been, it is impossible to say; perhaps a partial transformation into metallic selenium, although at that temperature the transformation should have been complete if it had taken place at all; possibly a transformation into one of the red crystalline forms; or possibly merely an experimental variation. At any rate, the observation being of an exceptional character, is worth recording.

Electrification of the Vitreous Form.

Seebeck (1826) reports that a rod of selenium (no doubt the vitreous form) becomes negatively electrified when rubbed, though less strongly than sulphur.

Reiss (1845) confirmed this observation of Seebeck. Berzelius had been unable to observe this phenomenon, though he records in his "Lehrbuch" that it was noted by Bondsdorff.

My experiments showed that vitreous selenium, when freshly prepared by pouring the melted material on a cold stone slab, shows very marked electrification.

Refraction and Dispersion of Vitreous Selenium.

These properties were investigated by Sirks (1871) who found the following values—

n _A	n _B	n _C	n _C	n _D
2.653	2.691	2.730	2.786	2.857
				2.98

Thus the element shows strong dispersive power, as well as very high refraction.

Diathermancy.

Schultz-Sellack (1869) furnishes the following data for the diathermancy of vitreous selenium—

Thickness of the selenium plate. M.m.	Percentage of heat passed through—	
	From lamp-black at 100°.	From an illuminating gas flame.
0.4	50	36
3.0	16	5

(To be continued).

ON THE FILTERING VALUE OF OLD ALLUVIAL DEPOSITS.

By Dr. F. MALMÉJAC.

With a view to estimating the filtering value of old and new alluvial deposits, we have examined the water borne by these different formations, and here give our first results.

The water examined came from the underground reservoir in the old gravels of Meurthe-et-Moselle. It was pumped up from a well sunk in the midst of highly cultivated land, which was manured every year. This well was 5 metres in depth, and the water level was 2 metres from the bottom. The samples were taken from a point intermediate between the surface of the water and the bottom. At the moment of taking each sample, the pump had been at work for twelve hours.

The first samples were taken before the application of manure, after a prolonged period of rain, snow, and thaw.

The last were taken four months after manuring with farmyard manure, during which months there had been on several occasions several days of rain followed by a long drought.

At the time of taking both samples, the pump was first made to deliver a large quantity of water, which then ran perfectly clear and limpid. The analyses of these samples for the two periods have given the following results:—

		Before manuring.	After manuring.
Hardness	Total	14°	15.5°
	Permanent	2°	1°
Residue at 100°		280	330
Colour of residue		Reddish	Grey
Sulphates, as H ₂ SO ₄		0	Distinct traces
Chlorides, as NaCl		0	Do.
Nitrates, as HNO ₃		10	Traces
Nitrites		0	0
Lime		110	145
Magnesia		0	Traces
Silica		11	Do.
Peroxide of iron		Traces	2
Organic matter in	Alkaline	1	5.0
m.grms. of oxygen			
per litre	Acid ..	1	5.2
Oxygen		10	7.4
Free ammonia		0	0.32
Albumenoid ammonia		Traces	0.40

The above results are expressed in m.grms. per litre.

These analyses show that, before the manuring, the water examined was quite fit for drinking purposes; after manuring, it had to be condemned.

A single analysis, however, is not sufficient on which to form an opinion of the fitness of a water for human use; before deciding definitely, it is necessary to examine several samples taken at different times.

The old alluvial deposits of Meurthe-et-Moselle, and probably all other alluvials of the same date, are, however, very bad filters, even in a thickness of 5 metres; not only do they allow organic matter to pass, but also all kinds of bacteria, as was conclusively shown by Dr. Imbeaux in his thesis on the potable waters of Meurthe-et-Moselle (Nancy, 1897).

In the case now under consideration, it is undeniable that the gravels have allowed the organic matter from the manure spread over the surface to reach the underground water.

This contamination is manifest by the considerable increase in the organic matter—the animal nature of which cannot be denied when we take into account the increase in the lime, sulphates, and chlorides, and, above all, the free and albumenoid ammonia which accompany it—while at the same time we see a diminution in the oxygen and in the nitrification.

Experiments now going on, and which will be published in due course, enable us to foresee that modern alluvial deposits, of the same thickness of 5 metres, are still worse filters than the older gravels.

It is therefore necessary whenever we have to pronounce an opinion on the value of a water for drinking purposes, to be extremely careful, when the supply is from alluvial gravels, if we have not had an opportunity of properly studying the surroundings of the source of supply.

We are, however, of the opinion that, whenever possible, it would be as well not to use water direct from alluvial gravels, as we have just shown that, with a thickness of 5 metres, this material is of no value whatever as a filter. —*Journ. de Pharm. et de Chim.*, Series 6, vol. xii., No. 8.

ANALYSIS OF THE WATERS FROM THE HOT SPRINGS AT DJEBEL ACHKEL.

By M. PUAUX.

At the foot of the Djebel Achkel, on the border of Lake Achkel, connected with the Lake of Bizerta by means of the Tindja, are three baths supplied by hot springs. During that period of the year when the region is accessible, these baths are much frequented by the Arabs.

The installation of these baths is of rudimentary simplicity. A natural basin, of about 4 metres area and 1.4 metres in depth, is covered over by the interlaced branches of the surrounding trees; the water rises naturally from the bottom of each basin and runs away over the edges. This bower is entered by an opening 1.3 metres in height.

The Arabs use these waters for the treatment of rheumatic affections, for which purpose they make superficial incisions in their limbs and then plunge them several times a day into the water. They also use these baths for the treatment of skin diseases, fevers, and probably all the ills their flesh is heir to.

These three baths are named as follows:—

Hammam *Sidi-bel-Abbés*, with a temperature of 45°; Hammam *Ain-Khalifif*, with a temperature of 43.5°; and Hammam *El-Djerab*, with a temperature of 43°.

The waters are all three limpid, and only have a little organic detritus floating on their surface, due to insufficient protection.

Chemical analysis shows that the composition of these three springs is identical, and that they come from a common subterranean source.

Composition of these Waters.

Density at 20°	1010
Residue dried at 120°	13.210
Residue heated to red heat ..	12.885
Sulphuric acid	1.907
Chlorine	5.515
Carbonic acid	0.098
Lime	1.707
Magnesia	0.780
Soda	2.973
Potash	0.270

The total weight of the different elements estimated is 12.85 grms., which agrees very closely with the residue heated to red heat.

The probable grouping of these elements is—

Sulphate of potash	0.499
Sulphate of magnesia	1.140
Sulphate of lime	1.560
Carbonate of lime	1.221
Chloride of lime	1.866
Chloride of sodium	7.563

A fourth spring actually rises under the waters of the

lake. We propose making an examination of this water in the course of a month or two.

These analyses lead us to the conclusion that the waters contain principally sodium chloride, and have a great analogy with those of the *Hammam-Lif* and *Bourbonne-les-Bains*.—*Journ. de Pharm. et de Chim.*, Series 6, vol. xii., No. 6.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, November 15th, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

Messrs. F. C. Britten, G. C. Thomson, R. G. Halstead, and J. A. Mathews were formally admitted Fellows of the Society.

The following certificates were read for the first time:—Messrs. H. H. Cousins, Government Laboratories, Kingston, Jamaica; Robert Dodd, 28, Sibella Road, Clapham, S.W.; Arthur Houldershaw, 71, Lavender Gardens, West Jesmond, Newcastle-on-Tyne; Edward Hughes, Redfory, Barmouth; Robert Salmon Hutton, Owens College, Manchester; Stephen Archigenes Ionides, Balliol College, Oxford; Robert Henry Jones, Glen Albyn, Coity Road, Bridgend, Glam.; H. T. G. van der Linde, 101, Tyndall Avenue, Toronto, Canada; William Meredith, 63, Albion Place, Ulverston; James Moir, The Ash, 62, Hamilton Place, Aberdeen, Scotland; H. M. Smith, 79, Helix Road, Brixton, S.W.

Of the following papers, those marked * were read:—

*147. "Trichlorobenzoic Acid." By FRANCIS EDWARD MATTHEWS.

The hexachloride of benzonitrile is acted upon by alcoholic sodium hydroxide, forming a mixture of trichlorobenzoic acids, which, on fractional crystallisation of the barium salts, was found to give a new trichlorobenzoic acid.

At present four of the six possible trichlorobenzoic acids are known, the missing two being—



As the new acid was found to yield an ester readily when treated with hydrogen chloride and alcohol, which an acid having the second of the above formulæ should not do, both positions adjacent to the carboxyl group being occupied by chlorine, the former of the two formulæ may be taken as proved.

By the action of quinoline upon the nitrile, $\text{C}_6\text{H}_5\text{Cl}_6\text{CN}$, followed by steam distillation; the nitrile of the new acid, $\text{C}_6\text{H}_2\text{Cl}_3\text{CN}$, is easily prepared in a state of purity. It crystallises from aqueous alcohol in long, white, lustrous needles melting at 87° . After hydrolysis with alcoholic sodium hydroxide, the acid, which crystallises from water in small needles melting at 163° , is readily obtained.

The following salts have been prepared:— $(\text{C}_6\text{H}_2\text{Cl}_3\text{CO}_2)_2\text{Ba} + 3\text{H}_2\text{O}$; $(\text{C}_6\text{H}_2\text{Cl}_3\text{CO}_2)_2\text{Sr} + 3\text{H}_2\text{O}$; $(\text{C}_6\text{H}_2\text{Cl}_3\text{CO}_2)_2\text{Ca} + 4\text{H}_2\text{O}$; $\text{C}_6\text{H}_2\text{Cl}_3\text{CO}_2\text{Ag}$. The acid chloride, $\text{C}_6\text{H}_2\text{Cl}_3\text{COCl}$, melts at 36° ; the amide, $\text{C}_6\text{H}_2\text{Cl}_3\text{CONH}_2$, crystallises in small needles which melt at $204\text{--}205^\circ$.

The ethyl ester, $\text{C}_6\text{H}_2\text{Cl}_3\text{CO}_2\text{Et}$, is obtained by the action of hydrogen chloride and alcohol upon the acid. It is a liquid volatile with steam, and possessing, but in a lesser degree, the odour of ethyl benzoate.

*148. "Oxidation of Benzalthiosemicarbazone." By GEORGE YOUNG, Ph.D., and WILLIAM EYRE, B.Sc.

Benzalthiosemicarbazone, $\text{C}_6\text{H}_5\text{CH:N:N:C(SH)NH}_2$, is oxidised by ferric chloride to *amidophenylthiodiazole*—



The base melts at $222\text{--}223^\circ$, its hydrochloride at $213\text{--}214^\circ$. The methyl derivative, $\text{C}_6\text{H}_5\text{C}_2\text{H}_5\text{SCH}_2\text{NH}_2$, is an oil; its platinichloride, $[\text{C}_6\text{H}_5\text{N}_2\text{S}]_2\text{H}_2\text{PtCl}_6$, melts at 218° . The acetyl derivative, $\text{C}_6\text{H}_5\text{C}_2\text{H}_5\text{SNHCOCH}_3$, which melts at 276° , is an acid forming stable sodium and silver salts. Both the methyl and acetyl derivatives yield the same methylacetyl derivative, $\text{C}_6\text{H}_5\text{C}_2\text{H}_5\text{SCH}_2\text{NCOCH}_3$, which melts at 144° .

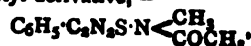
Benzal-4-methylthiosemicarbazone,—



is similarly oxidised to—

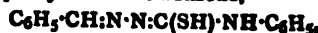


methylamidophenylthiodiazole, which melts at $183\text{--}184^\circ$. Its platinichloride, $[\text{C}_6\text{H}_5\text{N}_2\text{S}]_2\text{H}_2\text{PtCl}_6$, melts at $208\text{--}209^\circ$. The acetyl derivative,—



melts at 195° .

Benzal-4-phenylthiosemicarbazone,—



yields phenylamidophenylthiodiazole,—



previously described by Marchwald. Its silver derivative, $\text{C}_{14}\text{H}_{10}\text{N}_2\text{SAg}$, decomposes with explosive violence when suddenly heated.

*149. "The Nitration of Benzeneazosalicylic Acid." By J. T. HEWITT and J. J. FOX.

One of the authors has already pointed out (Hewitt, *Trans.*, 1900, lxxvii., 99) that when dilute nitric acid is warmed with benzeneazophenol a substance results in which the nitro-group has entered the phenol nucleus in the ortho-position relatively to the hydroxyl. No melting, on the other hand, by nitrating benzeneazophenol in strong sulphuric acid, obtained paranitrobenzeneazophenol (*Ber.*, 1887, xx., 2997).

The authors have studied the action of nitric acid on benzeneazosalicylic acid under varying conditions, and find that with dilute acid at $65\text{--}70^\circ$ the nitro-group enters the salicylic residue in the ortho-position relatively to the hydroxyl. The constitution of the resulting compound was determined by comparison with the product obtained from phenyldiazonium chloride and nitrosalicylic acid ($\text{CO}_2\text{H}:\text{OH}:\text{NO}_2=1:2:3$).

Benzeneazoorthonitrosalicylic acid melts at 197° (uncorr.), its methyl ester at $132\text{--}134^\circ$, and the ethyl ester at $128\text{--}129^\circ$.

By the nitration of benzeneazosalicylic acid dissolved in concentrated sulphuric acid, paranitrobenzeneazosalicylic acid, identical with the substance described by Meldola (*Trans.*, 1885, xlvii., 666), was obtained. Its ethyl ester melts at $220\text{--}225^\circ$.

*150. "Upon the Collection and Examination of the Gases produced by Bacteria from certain Media." By WALTER C. C. PAKES and WALTER H. JOLLYMAN.

The authors described a new apparatus for the collection of the gases produced by bacteria when grown either under aerobic or anaerobic conditions.

Experimenting with the *Bacillus pyocyaneus*, which is supposed to be a strictly aerobic organism, they found that it grew in media containing 1 per cent of potassium or ammonium nitrate under the strictest anaerobic conditions as the term is at present understood (that is, in the presence of hydrogen, or in the absence of any gas). They concluded, therefore, that the terms aerobic and anaerobic must be extended to include the presence of oxygen in the form of nitrates.

Upon analysing the gases produced by this organism from media containing nitrates, they found that both free oxygen and free nitrogen were evolved, the former in small quantities, but constantly.

151. "The Bases contained in Scottish Shale Oil." By FREDERIC CHARLES GARRETT, M.Sc., and JOHN ARMSTRONG SMYTHE, B.Sc., Ph.D.

Though many investigators have examined the bases contained in coal-tar, but little attention has been paid to those present in shale oil; the authors have therefore undertaken the examination of those obtained from the Broxburn oil. The shale, on distillation, yields gas, ammonia water, and "crude oil"; this "crude oil" is again distilled, giving "green naphtha," "green oil," and "still coke," the temperature towards the end of the operation rising to a red heat. A quantity of this "green naphtha" was washed with a $\frac{1}{2}$ per cent of sulphuric acid (diluted to about 15 per cent). Steam was blown through the reddish-brown liquid so obtained for several hours, to free it from a small quantity (about 0.1 per cent) of an oily liquid possessing a peculiarly disgusting smell; when this had been removed, sodium hydroxide solution (17½ per cent) was added, and superheated steam blown through until all the volatile bases had been driven over. The soluble bases in the distillate were thrown out by the addition of solid sodium hydroxide, the mixed bases were then dried by solid potash and fractionally distilled; each litre of the crude acid liquor yields about 120 grms. of the dry bases, of which about 25 per cent boil below 170°, and 46 per cent between 170° and 205°.

The basic oil thus obtained was then repeatedly refractionated, and an attempt made to isolate the various bases in the lower fractions by precipitation with mercuric chloride and fractional crystallisation of the salts so obtained. The portion boiling below 125° (about 10 grms.) was examined for pyridine by precipitation with potassium ferrocyanide, but none was found, the base being chiefly α -picoline (b.p. 128°); from the other fractions below 160° the symmetrical collidine ($\alpha\gamma\alpha$ -trimethyl pyridine) and two other bases which appear to be the hitherto undescribed lutidines ($\alpha\beta$ - and $\alpha\delta$ -dimethyl pyridine). The first of these boils at about 160° and yields a platinumchloride melting with decomposition at 208°, whilst the second boils at about 140°, and although no platinum compound was isolated it yielded a gold compound melting at 73°; in each case, however, the amount of base obtained was very small. Having now obtained between 2 and 3 kilogrammes of the raw bases, the authors are attempting a more systematic examination of them.

The authors have to thank Mr. George Beilby for calling their attention to the necessity for this work, and Mr. D. R. Steuart for kindly providing them with the raw material they required.

152. "On a Simplified Method for the Spectrographic Analysis of Minerals." By WALTER NOEL HARTLEY, F.R.S., and HUGH RAMAGE, A.R.C.Sc.I.

The authors pointed out that some of the rare earths are very difficult to detect, and others more widely diffused are not easily separated and recognised either by the ordinary methods of chemical analysis or by spectroscopic examination. A simplification of the method of obtaining oxyhydrogen blowpipe spectra (Hartley, *Phil. Trans.*, 1894, clxxxv., 168) was described and examples given of its application to the analysis of various metallurgical products, metallic ores, and siliceous minerals. The method is to burn the metals, minerals, or precipitates, on ashless filter-papers in an oxyhydrogen flame, and to photograph the spectra in the usual way. Refractory silicates in very fine powder are decomposed by being heated with strong sulphuric acid and ammonium fluoride which has been purified by distillation in a platinum retort. After the complete decomposition of the mineral, the sparingly soluble sulphates are separated by filtration and burnt on the ashless paper. The hydroxides of the heavy metals are precipitated by ammonia and similarly burnt.

The alkalis are contained in the aqueous solution of which the treatment is varied according to the nature of the substance under examination. For the separation of rubidium and cesium, platinic chloride is used and the platinum-chlorides collected and burnt.

Rammelsberg Memorial Lecture.

The Rammelsberg Memorial Lecture will be delivered by Professor H. A. Miers, D.Sc., F.R.S., on Thursday, December 13th, 1900, at 8.30 p.m.

NOTICES OF BOOKS.

Thirty-sixth Annual Report on Alkali, &c., Works. By THE CHIEF INSPECTOR. London: Eyre and Spottiswoode. 1900. Pp. 184.

THIS Report is on the Proceedings under the Alkali, &c., Works Regulation Acts during the year 1899.

The number of Works now registered under the Acts in England, Ireland, and Wales, is 1055. Of these 88 only are works decomposing salt, while the remainder carry on processes which are scheduled under the Acts of 1881 and 1892.

These numbers show a net increase of two alkali and one other works since the previous year. There are also 127 works registered in Scotland, bringing the total number up to 1182.

With regard to the escape of acid gases, two changes have been made; the first is to bring the statistics presented more into conformity with the wording of the Act of 1881, viz., the amount of hydrochloric acid condensed in place of that escaping by the chimney, is presented. Section 3 (a) of this Act provides for the condensation "of the muriatic acid evolved in such work, to the extent of 95 per cent," &c. The figures for 1897, 1898, and 1899 show 98.39, 98.53, and 98.60 per cent condensation respectively. The second change is an addition of the average escape of hydrochloric acid in salt-works. In 1884 the escape of hydrochloric acid in the extraction of salt from brine was put under the same limits of escape by the chimney as in alkali works, viz., 0.30 grain per cubic foot of chimney gases; it is therefore considered right to present this average figure annually in the table.

There have been 5011 visits and 5585 tests made during the year, and four prosecutions; though this latter figure is higher than usual, it can by no means be considered excessive, and we are glad to see that in the larger works there has been this year very little cause for anxiety, and much cause for praise in the efficiency of plant, and in the control attained over local escapes, as well as over those where the limits are prescribed by the Act.

The activity which has been so noticeable in the Leblanc process during the year 1899 has, of course, extended to the production of bleaching-powder and chlorates, and there has also been an increase in the production of chlorine by electrolytic processes.

The most striking event of the year 1899, in connection with the Leblanc industry, was the terrible explosion of 156 tons of chlorate of potash at the Kurtz works of the United Alkali Company, on May 12. By this explosion five men lost their lives, and a number of persons were injured. A special inquiry was held by Colonel Ford, C.B., into the circumstances attending the explosion, and his report has been issued as a Parliamentary paper. A curious and interesting effect of this explosion was noticed on the barometer at the Hardshaw Brook Chemical Works, about a quarter of a mile away; the mercury "suddenly rose, and immediately fell to a point indicated by some scum on the surface of the mercury being left behind in the barometer tube on the mercury rising again to its normal height. This point was at 22.60 inches from the surface of the mercury in the cup (corrected

indicating the vacuum created in the atmosphere after the explosion. The first effect of the explosion was a pressure which drove the mercury up above the normal height 2½ inches, until it was arrested by the closed end of the tube; . . . it then fell to 22·60 inches, and left the mark on the barometer tube mentioned. All the windows at Hardshaw Brook Chemical Works were blown inwards, but at a greater distance from the scene of the explosion the windows were blown outwards."

The Elements of Inorganic Chemistry, for Use in Schools and Colleges. By W. A. SHENSTONE, F.R.S. London: Edward Arnold. 1900. Pp. 506.

THE author's endeavour, in compiling this volume, was to provide a book which begins with experimental work for quite young students, and develops in the later stages into a text-book suitable for the older ones. The subject matter has been divided into numbered sections, which is not only useful for purposes of reference, but also enables the teacher to modify the course to the abilities of his various pupils and the time at their disposal.

The sections on carbon include a good deal that is usually omitted from books on inorganic chemistry, and there is a large number of references; these, if properly used, will of course be of considerable advantage, but most students have not the run of a good library, even if they have the inclination to use it.

The general arrangement of the book is good, and should help the teacher to lay a solid foundation in the minds of his pupils, ready for the additional load of more advanced work.

A Systematic Handbook of Volumetric Analysis; or the Quantitative Estimation of Chemical Substances by Measure, applied to Liquids, Solids, and Gases. By FRANCIS SUTTON, F.I.C., F.C.S. Eighth Edition, Enlarged and Improved. London: J. and A. Churchill. 1900. Pp. 640.

As speed is a very important factor in technical chemical estimations, it naturally follows that the use of volumetric methods should become more and more developed; many new processes have been made known since the publication of the last edition of this book, and this fact has caused some delay in the issue of the present edition, owing to the time it takes to find whether there is any real value in the new methods or in the modifications of the old ones; an endeavour has, however, been made to insert only those which have been actually tested and found to be of real use.

The general plan of the book is the same as in previous editions, but there are of course many important additions; for instance, as an Appendix to Part V, the author gives two new methods for the estimation of potash. The first was devised quite recently, during the printing of this book, and too late for notice in its proper place. This method is based on the fact that potash may be combined in an insoluble form as cobalti-nitrite of sodium and potassium, $K_2NaCo(HO_2)6H_2O$, and the amount of potash is found by using standard potassium permanganate as an oxidising agent, 1 c.c. of strictly N/10 permanganate being equal to 0·0007833 grm. of K_2O ; the author's experience is that with careful management very fair results may be obtained. The second method referred to is based on the formation of the double thiosulphate of bismuth and potassium and its estimation by iodine. Among the drawbacks to the method are the highly concentrated solution of the potash required and the large amount of pure alcohol necessary to be used. The end-point is somewhat difficult to distinguish, and care is necessary to procure pure, fresh, calcium thiosulphate.

These are but an example of many other new processes and methods which have been added to the book in order that it may keep its well-known place as the standard work on volumetric analysis, a place it took nearly forty

years ago, when Mr. Sutton brought out the first edition in 1863. Since that time many striking improvements have been made in chemical technology, but as the years have rolled on new editions have kept pace with discovery, while the reputation of *trustworthiness* has always been maintained.

A School Chemistry. By JOHN WADDELL, B.A. (Dal. Coll.), B.Sc. (Lond.), &c. New York: The Macmillan Co. London: Macmillan and Co. 1900. Pp. 278.

ALTHOUGH differing widely in style from the ordinary chemistry-book for beginners, we are of opinion that this volume will be favourably received. The subject is treated in a more popular manner than young students are accustomed to meet with, and it naturally follows that, when their interest is aroused, the lectures and laboratory work are more likely to be of lasting good. Water is first discussed, as being one of the most common substances, though, according to some modern writers, even the best water is only "diluted sewage"; then follows the consideration of hydrogen and oxygen, the latter leading to the study of air and its constituents. No mention of the atomic theory is introduced until the study of a large number of facts has afforded an intelligible basis for it, until, indeed, the pupil has in his possession most of the facts upon which Dalton founded the theory; he is thus enabled to obtain an accurate view of the real meaning of formulæ, and power to use them correctly. In other words, in this volume theory is subordinated to facts.

There is one point to which already we have drawn frequent attention in other books of this class, and that is the inappropriate nature of many of the illustrations; for instance, on page 173, we read "Experiment 79.—Place a piece of filter-paper upon a tripod as in the figure (Fig. 52)," and by the side there is a figure of a tripod with a piece of paper on it; what can be more unnecessary? Then, going to another extreme, in Chapter XIX., on the "Iron Group of Metals," we find illustrations of the sections of a blast-furnace and a reverberatory furnace, as well as a Nasmyth hammer and a Bessemer converter; greater discrimination should be exercised in this matter.

We note, on p. xiii., the names of several American firms recommended for supplying the apparatus needed for carrying out the experiments described in this book. Considering the book is published in London as well as in New York, we think some English firms might also have been mentioned.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

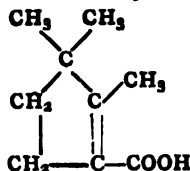
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxii., No. 20, November 12, 1900.

Chemical Reactions provoked by the Silent Electric Discharge.—M. Berthelot.—The author studies the subject of silent electric discharge with regard to its chemical effect under three different sets of conditions:—1. When such a current as is developed in a gaseous layer between two surfaces of dielectric bodies is used, influenced either by the variations of potential which determine the discharge of an induction coil or an electric machine, or by the constant potential difference between the two poles of an open electric pile. 2. When atmospheric electricity is considered, such as that existing under normal conditions, apart from storms and their explosive phenomena; that is to say, the differences in potential which exist between the different layers of air or between a layer of air and

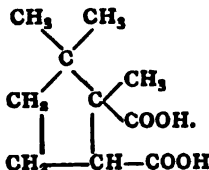
bodies situated on the earth's surface. 3. When electricity developed by inequalities of temperature is used, and that produced by chemical reactions giving rise to potential differences in the different regions of a gaseous system, constituted either of a single gas or of a mixture; or, further, between this gaseous system and liquid or solid bodies in contact with it.

Decomposition by Alkalis of Acetylenic Acetones.—Ch. Moureu and R. Delange.—A preliminary conclusion can be drawn from the experiments described in the present paper and in the authors' former paper, that, generally, boiling solutions of alkalis decompose acetylenic acetones. As to the method of decomposition, it may be remarked that benzoylphenylacetylene, acetylenanthrylidene, and benzoylanthrylidene are decomposed into acid and ketonic products (a single acid and a single acetone in the case of benzoylphenylacetylene, on account of the symmetry of the corresponding β -diketone), while the decomposition of acetylenic acetones into acids and acetones appears to be the general rule, so that the decomposition of acetylphenylacetylene into phenylacetylene and acetic acid is an exception. The authors intend to prepare and investigate other acetylenic acetones, so as to determine, if possible, in what cases the decomposition into acetylenic and acid carbide takes place, which decomposition, up to the present, appears to be peculiar to acetylphenylacetylene.

Constitution of Camphoric Acid, and the Migrations which take place in the Molecule.—G. Blanc.—All preceding papers published by the author on the constitution of camphoric acid had for their aim the representation of isolaunonic acid by the formula—



As this acid is derived from camphoric acid by extraction of the elements of water and carbonic oxide, the formula of camphoric acid can easily be deduced, and is found to be—



Several objections have been raised to this formula. The author discusses and explains these objections.

Evolution of Terpenic Compounds in Geranium.—Eug. Charabot.—The author's experiments on geranium prove that the etherification effected is in proportion to the development of the plant. The transformation from geraniol into rhodinol is represented by the equation $\text{C}_{10}\text{H}_{18}\text{O} + 2\text{H} = \text{C}_{10}\text{H}_{20}\text{O}$, and takes place in the green parts of the plant, which, as is well known, constitute the chief reducing media. This hypothesis agrees with the chemical experiments which the author has made, and with the physiological facts already accepted.

Presence of Invertine or Sucrose in Grapes.—V. Martinand.—In the juice of all the species of grape which the author was able to procure, a decided quantity of invertine was found, which is able, under the conditions of his experiments, to invert a quantity of saccharose—almost double the quantity of original wort used. At 30°, the temperature of fermentation of grape wort, the increase of invert sugar is also considerably raised. However, the sucrose disappears completely in wines after being submitted to strong oxidation.

MISCELLANEOUS.

Royal Institution.—The following are the Lecture Arrangements at the Royal Institution, before Easter:—Sir Robert Ball, Six Lectures (adapted to young people) on "Great Chapters from the Book of Nature."

Professor J. A. Ewing, Six Lectures on "Practical Mechanics (experimentally treated), First Principles and Modern Illustrations."

Dr. Allen Macfadyen, Fullerian Professor of Physiology, R.I., Four Lectures on "The Cell as the Unit of Life."

Dr. Arthur Willey, Three Lectures on "The Origin of Vertebrate Animals."

The Rev. H. G. Graham, Three Lectures on "Society in France before the Revolution."

Professor Percy Gardner, Three Lectures.

Sir Wyke Bayliss, Two Lectures on "Shakespeare in Relation to his Contemporaries in Art."

Professor R. K. Douglas, Two Lectures on "Government and People of China."

Mr. F. Corder, Three Lectures on "Vocal Music, its Growth and Decay" (with Musical Illustrations).

The Right Hon. Lord Rayleigh, Six Lectures on "Sound and Vibrations."

The Friday Evening Meetings will begin on January 18th, when a Discourse will be delivered by Professor Dewar on "Gases at the Beginning and End of the Century." Succeeding Discourses will probably be given by Dr. A. W. Ward (the Master of Peterhouse), the Right Rev. Monsignor Gerald Molloy, Professor G. H. Bryan, Professor J. J. Thomson, Sir W. Roberts-Austen, Mr. H. Hardinge Cunyngame, Mr. W. A. Shenstone, Dr. Horace Brown, The Right Hon. Lord Rayleigh, and other gentlemen.

On Tungsten. Preparation of Tungsten with the Help of Liquid Air.—A. Stavenhagen.—To obtain the highest possible temperature we proceed in the manner already described (*Berichte*, xxii., p. 1413; *CHEM. NEWS*, lxxi., p. 227), but also add to the mixture in the crucible one-third of its volume of liquid air; the mass is well stirred and put on the fire; it is as well not to work on more than 50 grms. of material. The temperature soon rises nearly to a white heat, and a well-melted ingot of tungsten is obtained nearly free from aluminium. The electrolysis of paratungstate of lithium, conducted in a porcelain crucible at 1000°, with a current of 3 amperes at 15 volts, after washing the mass with water, hydrochloric acid, and a boiling solution of lithia, gives crystals of tungsten containing about 6 per cent of aluminium. But with a current of 3.5 amperes and 12 volts we obtain a tungsten-lithium bronze in fairly large bluish-black crystals; this has already been prepared by H. Scheibler. —*Berichte*, xxii., p. 3064.

Preparation of Molybdenum and Uranium with the Help of Liquid Air.—A. Stavenhagen.—We proceed as in the case of tungsten (see preceding paragraph). With molybdenum the return is low, on account of the volatility of MoO_3 . With uranium we notice that the mixture $\text{UO}_3 + 2\text{Al}$, with a slight excess of Al, burns with difficulty by itself, and the uranium does not unite in an ingot. On the addition of liquid air (20 grms. for 30 grms. of the mixture), the deflagration is very violent, accompanied by a brilliant light, and an ingot of uranium is then obtained. —*Berichte*, xxii., p. 3065.

The Compounds of Basic with Acid Colouring Materials.—A. Seyewetz.—The compounds of basic with acid colouring materials are remarkable for their insolubility in water, which appears to be the greater as the colours used have a more marked acid or basic character. This property can thus be used, in certain cases, as a delicate qualitative reaction, as in very dilute solutions a precipitate will be formed by the addition of another suitable colouring-matter. —*Bull. de la Soc. Chim. de Paris*, Series 3, xxiii., No. 12.

MEETINGS FOR THE WEEK.

- MONDAY, 10th.—Society of Arts, 8. (Cantor Lectures). "Electric Oscillations and Electric Waves," by Prof. John A. Fleming, M.A., D.Sc., F.R.S.
- WEDNESDAY, 12th.—Society of Arts, 8. "The Treatment of London Sewage," by Prof. Frank Clowes, D.Sc.
- THURSDAY, 13th.—Chemical, 8.30. "Rammelsberg Memorial Lecture," by Prof. H. A. Miers, D.Sc., F.R.S.
- FRIDAY, 14th.—Physical, 5. (This meeting will be held in the Physical Laboratory of the Royal College of Science, South Kensington). "Electric Inertia" and "The Effect of Inertia on Electric Currents in a Rotating Sphere," by Prof. A. Schuster, F.R.S. "Exhibition and Description of a Quartz-thread Gravity Balance," by Prof. R. Threlfall, F.R.S. "On the Theory of Magnetic Disturbances by Earth Currents" and "The New Physical Laboratories of the Royal College of Science," by Prof. A. W. Rücker, F.R.S. "Notes on the Practical Application of the Theory of Magnetic Disturbances by Earth Currents," by Dr. R. T. Glazebrook, F.R.S. Exhibition of a Set of Half-seconds Pendulums, by W. Watson.

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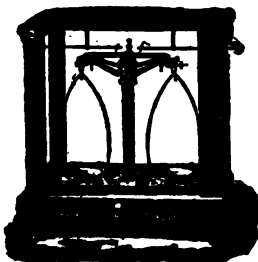
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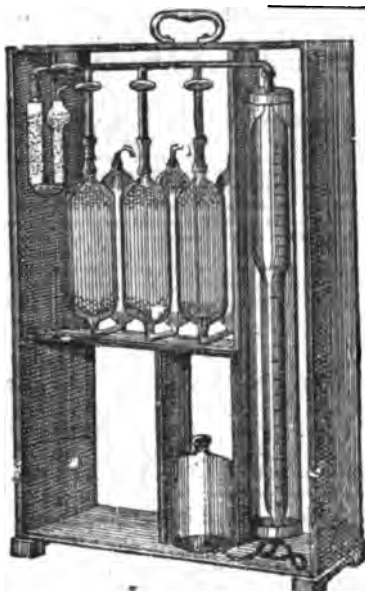
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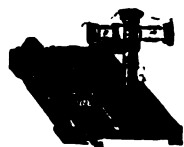
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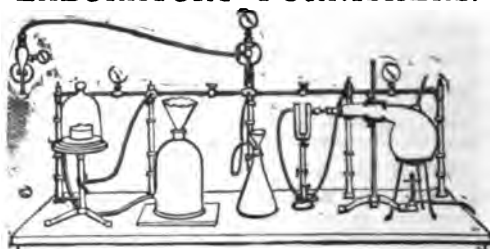
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(Continued from p. 260).

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I. OXIDATION TO SULPHURIC ACID, WITH—

- (a). Gravimetric Estimation.
- (b). Volumetric Estimation.

II. EVOLUTION AS SULPHURETTED HYDROGEN, WITH—

- (a). Gravimetric Estimation.
- (b). Volumetric Estimation.

III. MISCELLANEOUS ITEMS.

I. OXIDATION TO SULPHURIC ACID.

I.a. Gravimetric Estimation.—

- 764. LEFORT (C. N., xix., 132).—One part HCl to three parts HNO_3 is the most suitable proportion of these acids for the rapid oxidation of sulphur.
- 765. NICKLES (C. N., vii., 87).—The iron is dissolved in bromine; the S precipitated as BaSO_4 .
- 766. TOSH (C. N., xvi., 168).—Dissolve in HNO_3 with a little HCl, evaporate, re-dissolve in HCl, and add BaCl_2 .
- 767. EGGERTZ (C. N., xvii., 207).—To solution of KClO_3 containing the iron, add HCl, evaporate, filter, and add BaCl_2 to nearly neutral solution. A similar process for iron minerals containing either Ba, Sr, or Pb.
- 768. PIESSE (C. N., xxviii., 248).—Dissolve in aqua regia, evaporate nearly dry, take up in HCl, and add BaCl_2 . WILLIAMS (C. N., xxviii., 266).—Same process, but dissolves in 1:20 HNO_3 .
- 769. ALLEN (C. N., xxix., 91 and 112).—HCl retards, if it does not altogether prevent, the precipitation of BaSO_4 ; neutralise the iron solution as far as possible with ammonia, and put aside for twenty-four hours. (See 773).
- 770. PLATZ (Z. I. S. I., 1887, i., 471).—Dissolve in 1:18 HNO_3 , evaporate with HCl, &c. The BaSO_4 and wet filter are ignited at low temperature, and then more strongly to oxidise any sulphide to sulphate. (See 786).
- 771. REIS (Z. I. S. I., 1889, ii., 479).—When iron is dissolved in HNO_3 , sulphur escapes estimation as H_2S and S. (See 862).
- 772. PARRY and MORGAN (C. N., lxxvii., 247).—Dissolve in aqua regia, evaporate three or four times with HCl to expel HNO_3 , dilute faintly HCl solution to 700 c.c., and add BaCl_2 . BaSO_4 is soluble in acid Fe_2Cl_6 . Or evolve as H_2S , absorb in CuSO_4 , and

ignite precipitate to CuO (see 885). S may be retained by the H_2SO_4 liquor and the insoluble residue.

- 773. ARCHBUTT (Z. S. C. I., 1890, 25).—In the aqua regia process, BaSO_4 should be precipitated from the concentrated solution; precipitation is complete in two hours, and excess of HCl does no harm (see 841).
- 774. ANON. (Z. I. S. I., 1893, i., 410).—The nitro-hydrochloric process is subject to error, through formation of basic salts before filtering off SiO_2 or during precipitation of BaSO_4 , and precipitating BaSO_4 from too acid a solution.
- 775. BAMBER (Z. I. S. I., 1894, i., 319).—Dissolve metal in HNO_3 , add Na_2CO_3 short of alkalinity, evaporate, roast, extract with dilute Na_2CO_3 , and precipitate as BaSO_4 .
- 776. PHILLIPS (C. N., lxii., 239).—S in Cu is estimated by precipitating as BaSO_4 from CuCl_2 solution; BaSO_4 is soluble in $\text{Cu}(\text{NO}_3)_2$. Precautions taken against contamination of the BaSO_4 with AgCl or PbSO_4 .
- 777. BRUYN (Z. C. S., lxii., 753) and HEATH (Z. S. C. I., 1896, 218).—Dissolve Cu in HNO_3 , precipitate electrolytically, and estimate S in the Cu-free liquor.
- 778. HAMPE (Z. S. C. I., 1891, 486).—Estimate S in Pb by adding filings to molten KNO_3 , passing CO_2 through water solution to precipitate Pb, and adding BaNO_3 to the filtrate.
- 779. CAMPBELL (C. N., lvi., 74).—Boil soluble slag with HCl and Br, and precipitate nearly neutralised filtrate with BaCl_2 .
- 780. ATKINSON (Z. I. S. I., 1896, ii., 207).—Roast powdered ore, short of fusion, with Na_2CO_3 , raising the temperature gradually, dissolve out Na_2SO_4 , and precipitate as BaSO_4 .
- 781. PHILLIPS (C. N., lxxv., 194).—By fusion of powdered irons with Na_2O_2 a higher percentage is obtained than by oxidation with acids. The interference of organic sulphur compounds with evolution processes.
- 782. MEINECKE (C. N., lix., 107), BOUCHER (C. N., lxxiv., 76), and CARNOT and GOUTAL (Z. C. S., lxxii., ii., 520).—Decompose Fe with acid cuprammonium chloride at boiling-point, oxidise filtered residue with aqua regia and KClO_3 , or Br and HCl, and precipitate as BaSO_4 .
- 783. TAMM (Z. I. S. I., 1887, ii., 369).—On account of solubility of BaSO_4 in Fe_2Cl_6 , add BaCl_2 , evaporate, fuse impure BaSO_4 with Na_2CO_3 , and re-precipitate. Wiborgh's (925) is more accurate than Eggertz's (922) colour process.
- 784. REIS (Z. I. S. I., 1889, i., 396).—Heat metal with MgO and NaHO , leach, filter, and boil with BaCl_2 .
- 785. MEINECKE (Z. C. S., lxxvi., ii., 518).—Dissolve the pig in HCl and KClO_3 ; expel Cl, reduce Fe with Zn, and add BaCl_2 .
- 786. BOUSSINGAULT (C. N., xv., 324).— BaSO_4 (CaSO_4 , PbSO_4 , &c.) is decomposed at high temperatures, and therefore should not be unduly heated.
- 787. MARSH (C. N., lix., 309).—Appreciable amounts of BaSO_4 are reduced by the carbon of the filter-paper. Precipitate should be treated with H_2SO_4 , and again ignited.
- 788. RIPPER (Z. C. S., lxxiv., ii., 239).—Oxidises sulphate reduced by the paper with H_2SO_4 and Br, and removes other impurities by digesting with HCl and Br (see 880).
- 789. MARBOUTIN (C. N., lxxvi., 232).—By igniting BaSO_4 filter apex upwards no sulphides are formed.
- 790. FOULK (Z. C. S., lxxii., 189).—Acid solutions need an excess of BaCl_2 to completely precipitate the S. The moist filter is ignited apex up.
- 791. SLEEPER (C. N., lxxix., 63).— BaSO_4 does not carry SiO_2 down with it. SiO_2 can be volatilised from BaSO_4 if free H_2SO_4 is present also.

792. RICHARDS and PARKER (C. N., lxxii., 281).—The occlusion of BaCl_2 by BaSO_4 increases with concentration and the presence of HCl. Results corrected by estimating Cl in the precipitate and deducting equivalent amount of BaCl_2 .
793. SLOANE (C. N., xlv., 221).—The various means of purifying BaSO_4 precipitates, and objections to them.
794. ZISGLER (Y. I. S. I., 1882, 384).—Add AgNO_3 after BaCl_2 to facilitate and complete the precipitation of BaSO_4 ; wash filter with AmHO to remove AgCl .
795. JANNASCH and RICHARDS (C. N., lx., 19).— BaSO_4 is insoluble in cold ferric solutions. A portion of the iron is always precipitated as barium-ferric sulphate [Schneider, (Y. I. S. I., 1893, i., 409) disputes this], which is decomposed with loss of SO_3 on ignition. Organic acid does not prevent this contamination (see 803 and 846, and Richards, C. N., lxxii., 185).
796. AULICH (Y. I. S. I., 1899, ii., 486).—Résumé of the processes for preventing contamination of BaSO_4 with Fe.
797. MEINECKE (Y. I. S. I., 1899, ii., 430) and HEIDENREICH (Y. C. S., lxxvi., ii., 517).—To prevent iron accompanying BaSO_4 , reduce to FeO with zinc before precipitating.
798. HERTING (Y. C. S., lxxvi., ii., 804).—Reduce to FeO with SnCl_2 before adding BaCl_2 .
799. FRESSENIUS and HINTZ (C. N., lxxiii., 276).—Particulars of the solubility of BaSO_4 with excess of BaCl_2 or H_2SO_4 in water, alkaline chloride, HCl, and HNO_3 solutions.
800. MORGAN (C. N., lviii., 63 and 75).—Reviews some well known objections to the methods based on oxidation and precipitation as BaSO_4 and evolution as H_2S .
801. SPILLER (C. N., x., 219).—Glacial H_3PO_4 and pyrophosphate interfere with the precipitation of BaSO_4 .
802. CROSSLEY (C. N., v., 245).—When S is oxidised by evaporation with HNO_3 an alkaline salt must be added to prevent SO_3 being volatilised.
- 802a. J. T. (C. N., lxxviii., 294).—The blank of the acids used should be found by treating two samples (one half the weight of the other) in the usual way, and deducting the BaSO_4 of the larger from twice the BaSO_4 of the smaller sample.

(To be continued).

THE SPECTRUM ANALYSIS OF NEODYMIUM AND PRASEODYMIUM.

By W. MUTHMANN and L. STÜTZEL.

THE authors first discuss the question as to whether neodymium and praseodymium are really simple bodies, but without giving any definite opinion.

If these elements are provisionally looked upon as undecomposable, it must be remarked that their absorption spectra vary considerably with the dilution, and, above all, with the nature of the acid present. The results are particularly striking if we compare the spectra of the chlorides or the nitrates with those of the carboxylised acetates, lactates, or double alkaline carbonates: the spectra become unrecognisable from one series to the other. Thus, while the solution of chloride of neodymium is red, that of the double potassic carbonate is distinctly blue, and the absorption bands are entirely displaced. The authors have also made estimations of neodymium and praseodymium by examining the absorption bands of various solutions by means of Vierordt's spectro-photometric method; the concentration, c , is related to the intensity I

of a band, A being a constant, by the equation $c = \frac{A}{\log I}$. The agreement with chemical estimations is very satisfactory, especially for strengths of 2 to 6 per cent.

The application of this method to the analysis of didymiferous minerals of different kinds and sources, shows the curious fact that neodymium is always present in practically double the amount of praseodymium.—*Berichte*, vol. xxxii., p. 2653.

THE PRECIPITATION AND SEPARATION OF COPPER IN ALKALINE SODIC SOLUTION BY MEANS OF SULPHATE OR HYDROCHLORATE OF HYDRAZINE.

By PAUL JANNASCH and K. BIEDERMANN.

ONE of the writers has already been engaged with the quantitative precipitation of copper in the presence of large quantities of caustic soda or potash, with the object of applying it to a certain number of metallic separations. Up to the present time this has only been effected quantitatively with metals whose hyperoxides (peroxide of hydrogen method), or hydroxides are insoluble in an excess of soda. Such, for instance, is the case in the separation of arsenic, associated with manganese or iron (Jannasch, *Praktischer Leitfaden d. Gewichtsanalyse*, 65). This, however, is no longer the case with oxide of copper, which is slightly soluble in caustic soda. When we have to do with this metal, it is necessary, for accurate work, to get rid of the excess of alkali after precipitation.

The first exact experiments, carried out by one of the writers in collaboration with H. Winkler, were based on the use of hydroxylamine, which precipitates the whole of the copper in the state of cuprous oxide in strongly alkaline solutions. After having overcome several technical difficulties connected with the filtration of the precipitate, and the prevention of partial re-oxidation, the above mentioned chemists succeeded in obtaining good results, both in the estimation of copper alone and in the separation of this metal from zinc and arsenic.

As for the difficulties mentioned above, the authors of this present paper have entirely removed them by replacing the hydroxylamine with hydrazine of Curtius, who kindly placed some sulphate at their disposal.

I. Determination of Copper.

The quantitative precipitation of this metal is effected in alkaline solution in the presence of sulphate of hydrazine. To effect the estimation, we take an accurately weighed quantity of cupric sulphate and dissolve it in a beaker with a little warm water. The solution thus prepared is then run into a porcelain crucible containing pure soda lye (5 grms. NaOH : 50 c.c. H_2O). As the copper salt is added in the cold, a pale blue precipitate of hydrate of copper is first formed. At this moment a boiling solution of 3 per cent sulphate of hydrazine (1–2 c.c.) is added, and the whole then gently heated on the water-bath with continual stirring. There is an immediate disengagement of gas, and the formation of protoxide of copper, which, after the addition of a further quantity of the solution of hydrazine (about 3 c.c.), is converted into metallic copper. This latter is quickly deposited at the bottom of the beaker. It is necessary to allow the liquid to cool, or to dilute it with water before filtration, to prevent the alkali from attacking the paper. We may further make use of two filter-papers with advantage, having the interior one about a couple of centimetres shorter than the other; by this means we can prevent small particles of the substance from passing over the edge of the paper during the washing. The precipitate must be washed with warm water until the washings no longer react on litmus, and leave no residue when evaporated to dryness on a strip of platinum foil. It is then dried at 90° , the filter calcined apart in a tared porcelain crucible, and after having added the rest of the

precipitate, the whole is heated to redness in a current of oxygen. This latter condition is not absolutely necessary if we take care to stir the material with a platinum spatula so that every part of it may come in contact with the air. The calcination is continued until the weight is constant, and as a further precaution, we make sure that the transformation into cupric oxide is complete by treating the product of incineration with a few drops of concentrated nitric acid. The authors have from time to time made a control calcination in oxygen.

First Analysis.—0.2218 grm. $\text{CuSO}_4 + 5\text{H}_2\text{O}$: 0.0704 grm. CuO = 0.0562 grm. Cu = 25.29 per cent (theory = 25.42 per cent).

Second Analysis.—0.2094 grm. $\text{CuSO}_4 + 5\text{H}_2\text{O}$: 0.0666 grm. CuO = 0.0532 grm. Cu = 25.41 per cent (theory = 25.42 per cent).

II. Separation of Copper and Zinc.

The salts employed for this separation were sulphate of zinc and sulphate of copper, of which the aqueous solution was poured drop by drop into a vessel containing 10 per cent soda-lye. The use of glass must be avoided, as it resists the action of the alkaline solution of hydrazine very badly, thus causing the formation of impurities whose presence would interfere with the accuracy of the estimation. In some experiments even platinum crucibles were found to be unsuitable, as they often permitted particles of precipitated copper to adhere to their sides.

We then add 5 c.c. of a 3 per cent solution of hydrazine to the cold, strongly alkaline liquor, and heat carefully with continual stirring. After a few minutes the copper is completely precipitated in the state of a reddish-brown precipitate, which is treated in the manner described above. After prolonged washing with warm water, the last traces of hydrate of zinc can be removed by treating with very dilute, warm caustic soda. The authors, however, have hardly ever found this kind of impurity in the numerous estimations they have carried out. The copper, dried at 90°, is then calcined in a porcelain crucible until the weight is constant. It is necessary, as has been explained in the case of the estimation of copper alone, to disaggregate the mass with a platinum spatula. This operation may in the same way be replaced by treatment with oxygen or concentrated nitric acid.

As for the solution containing the zinc it is treated with hydrochloric acid until the precipitate first formed is re-dissolved, and the colourless liquid has a distinct acid reaction. We then add excess of a solution of carbonate of soda, stirring the whole time. To prevent loss by projection the beaker is covered with a watch-glass with a hole to allow the passage of the stirring rod. The carbonate of zinc is then converted into oxide by calcination, and this, if it does not contain any trace of silica, should be absolutely white, and should give a clear limpid solution with dilute acetic acid.

First Analysis.—0.2940 grm. $\text{CuSO}_4 + 5\text{H}_2\text{O} + 0.1525$ grm. $\text{ZnSO}_4 + 7\text{H}_2\text{O}$ = 0.4465 grm. substance; 0.0935 grm. CuO = 0.0746 grm. Cu = 16.73 per cent (theory = 16.77 per cent); and 0.0431 grm. ZnO = 0.3430 grm. Zn = 7.68 per cent (theory = 7.74 per cent).

Second Analysis.—0.3040 grm. $\text{CuSO}_4 + 5\text{H}_2\text{O} + 0.1890$ grm. $\text{ZnSO}_4 + 7\text{H}_2\text{O}$ = 0.4930 grm. substance; 0.0949 grm. CuO = 0.0770 grm. Cu = 15.61 per cent (theory = 15.69 per cent); and 0.0527 grm. ZnO = 0.4230 grm. Zn = 8.58 per cent (theory = 8.68 per cent).

III. Separation of Copper and Arsenic.

In this case the writers have used cupric sulphate and arsenious acid. This latter was transformed, for the purpose of estimation, into arsenic acid by the aid of a few drops of concentrated nitric acid.

The aqueous solution of the two elements to be separated, was gradually added to 40 or 50 c.c. of 10 per

cent soda-lye, and after the addition of 4 or 5 c.c. of the solution of sulphate of hydrazine, the mixture was submitted to the action of heat. In this manner a precipitate of metallic copper is formed which is treated as before.

The arseniate of soda passes into the filtrate, and is in its turn treated in the following manner:—For the purpose of first removing the excess of soda, the alkaline solution is saturated with hydrochloric acid, to which a small quantity of nitric acid is added at the end; the whole is then concentrated down to about 80 c.c. If the solution contains any silica from the glass of the apparatus, it is necessary to saturate with nitric acid, evaporate to dryness, take up the residue with acidulated water, and separate the insoluble impurities by filtration. When the filtrate is quite cool, we add to the clear, colourless solution an excess of ammonia and freshly prepared magnesia mixture. The mixture is then left at the ordinary temperature for fifteen or twenty-four hours, and we can then proceed with the filtration of the ammonio-magnesian arseniate, which is frequently deposited in the form of beautiful well-formed needles. (For further details refer to *Prakt. Leitfaden der Gewichtsanalyse*, p. 64).

First Analysis.—0.1255 grm. $\text{As}_2\text{O}_3 + 0.2801$ grm. $\text{CuSO}_4 + 5\text{H}_2\text{O}$ = 0.4056 grm. substance; 0.1964 grm. $\text{Mg}_2\text{As}_2\text{O}_7$ = 0.0948 grm. As = 23.37 per cent (theory = 23.41 per cent); and 0.0886 grm. CuO = 0.0708 grm. Cu = 17.46 per cent (theory = 17.54 per cent).

Second Analysis.—0.2230 grm. $\text{As}_2\text{O}_3 + 0.2556$ grm. $\text{CuSO}_4 + 5\text{H}_2\text{O}$ = 0.4786 grm. substance; 0.3495 grm. $\text{Mg}_2\text{As}_2\text{O}_7$ = 0.1687 grm. As = 35.24 per cent (theory = 35.29 per cent); and 0.0813 grm. CuO = 0.0649 grm. Cu = 13.56 per cent (theory = 13.68 per cent).

IV. Separation of Copper and Tin.

As the result of a long series of experiments and more than thirty estimations, the authors have succeeded in finding a practicable and exact method for the separation of these two metals. They used a mixture of pure copper and pure tin, which they dissolved in the quantity of aqua regia necessary to obtain a bright green solution. If an excess of these acids has been used it is necessary to get rid of it after the solution is complete, by means of careful evaporation. The solution diluted with water is then poured drop by drop into a solution of caustic soda containing 15 times the weight of alkali that there is of metal present, and to which 2 or 3 grms. of hydrochlorate of hydrazine have been added. The action of this latter salt is identical with that of the sulphate used in the preceding operations. However, after having made numerous separations of copper and tin the authors have come to the conclusion that the presence of sulphates interferes with the accuracy of the analysis. In fact, in such a case the copper always contains traces of tin which are impossible to remove by washing with warm dilute soda, and which therefore make the amount of copper found too high.

The precipitated copper is heated for some time in a porcelain crucible in the presence of excess of hydrazine so as to dissolve the small quantity of tin which has escaped the action of the alkali in the state of stannous stannate. After cooling and diluting the liquid, the precipitate is thrown on a double filter and carefully washed with boiling water. The rest of the treatment is the same as with the previous separations.

The alkaline solution containing the tin is then treated with a slight excess of concentrated hydrochloric acid and saturated with ammonia. This latter causes the precipitation of the stannous hydrate which is re-dissolved in sulphide of ammonium. This new solution is then heated for some time on the water-bath, and made slightly acid with dilute hydrochloric acid. The sulphide of tin which is deposited by this means is filtered, washed with a warm solution of sulphuretted hydrogen, and transformed into stannic acid by calcination in a current of oxygen.

First Analysis.—0.0813 grm. Cu + 0.2336 grm. Sn = 0.3149 grm. substance; 0.1014 grm. CuO = 0.0810 grm. Cu = 25.72 per cent (theory = 25.81 per cent); and 0.2065 grm. SnO₂ = 0.2332 grm. Sn = 74.06 per cent (theory = 74.18 per cent).

Second Analysis.—0.1247 grm. Cu + 0.2729 grm. Sn = 0.3976 grm. substance; 0.1558 grm. CuO = 0.1244 grm. Cu = 31.28 per cent (theory = 31.38 per cent); and 0.3461 grm. SnO₂ = 0.2723 grm. Sn = 68.48 per cent (theory = 68.61 per cent).

—*Berichte*, vol. xxxiii. p. 631.

FURTHER EXAMINATION OF THE SOLUBILITY OF LIME IN SACCHARINE SOLUTIONS.

By J. WEISBERG.

I. The Solubility of Lime in its Different Forms, in Saccharine Solutions at the Ordinary Temperature.

In a previous paper (*Bull. Soc. Chim.*, Series 3, vol. xxi., p. 773) we examined the solubility of powdered calcic oxide (CaO) in solutions of sugar at a temperature of 15–16°, and showed that this solubility is much greater than has been observed by other chemists who have already studied this question. But, as far as the extreme limit of saturation of the saccharine solutions experimented on is concerned, we added that we could not guarantee having absolutely reached it in our experiments. To attain the maximum limit of saturation, it is necessary to operate with a very large excess of lime compared with the amount which could be actually dissolved.

We now complete our previous research on this subject, by considering in this paper not only the solubility of the dry, powdered, calcic oxide, but also that of hydrate of lime, Ca(OH)₂, and of milk of lime.

To determine the comparative solubility of lime in its three different forms which are in practical use, the quantities of material added in excess in the three different cases were kept as nearly as possible in the same proportions. The saccharine solutions experimented upon were treated with practically identical quantities of lime in its three different forms; the stoppered flasks containing the sucro-calcic mixtures were left, with occasional agitation, as long as was necessary to obtain filtered solutions of constant composition. The temperature at which we carried out these experiments varied only between very narrow limits (15–16°). All the results of these comparative experiments are given in Table I. in full detail. The figures in that table show:—

1. That the solubility of lime in its three forms, in saccharine solutions of known strength and at a temperature of 15–16°, is much greater than has been recorded by previous observers.

2. That, under the same experimental conditions, oxide of calcium is the most soluble, followed by hydrate of lime and then by milk of lime, in which form the relative solubility of lime is the weakest.

As far as concerns oxide of calcium, its solubility in saccharine solutions of medium concentration is about 28 parts of CaO for every 100 parts of sugar in solution; but this solubility, although great, does not yet represent the extreme limit, for, on different occasions, we have prepared sucro-calcic solutions in which we have estimated the presence of 28.5, 29.0, 29.5, and even 30.5 (the extreme limit) parts of CaO for each 100 parts of sugar in solution, and that, in solutions the concentration of which varied between 8 and 17 per cent of sugar, using powdered calcined marble, or again, a well-burnt lime from a very pure commercial carbonate of lime, and at a temperature of 15–16°.

If our table does not contain these figures of high solubility, it is because we did not admit any but *stable* sucro-

calcic solutions—that is to say, solutions which remained clear even when the temperature was lowered several degrees relatively to that at which their composition was determined. The sucro-calcic solutions containing 28.5 to 30 parts of CaO for each 100 parts of sugar (saccharine solutions containing 10 to 16 per cent of sugar), at a temperature of 16°, do not always remain limpid when left at a temperature lower than that we have mentioned; they often give a deposit more or less voluminous, and on separating the supernatant liquid from the deposit we find in the filtrate a quantity of dissolved lime equivalent to that given in the table (27 to 28).

All the sucro-calcic solutions experimented upon, when heated on the water-bath, became cloudy, gave a deposit, and, according to the concentration, became gelatinous, but the deposit formed during the heating was re-dissolved on cooling, and the solutions became clear and limpid again. The more lime the liquid contained, the sooner the gelatinous precipitate commenced to form on heating. Of two saturated saccharine solutions of lime, heated simultaneously on the water-bath, it is the solution which has its lime in the form of oxide of calcium which becomes cloudy first, while the other sucro-calcic solution which has its lime in the form of hydrate, or of milk of lime, does not become cloudy until a little later, when the temperature has increased to several degrees higher than the first one. Sucro-calcic solutions of medium concentration (containing 10 to 16 per cent of sugar and 27 to 28 parts of lime for each 100 parts of sugar in solution), when heated in a test-tube on the water-bath, first become cloudy, and then form a white paste sufficiently solid for the tube to be turned upside down without any material falling out.* On cooling, the paste becomes liquid again, and the solution is as clear and limpid as before heating.

II. On the Solubility of Lime in Saccharine Solutions at a High Temperature.

Dubrunfaut (*Comptes Rendus*, xxxii., p. 498) and Lippmann ("Chemie der Zuckurarten," p. 620), first observed that the solubility of lime in solutions of sugar, as well as in pure water, depends essentially on the temperature; but it is to Lamy ("La Sucrerie Indigène et Coloniale," vol. xi., p. 234-237), and Lippmann ("Chemie der Zuckerarten," p. 650), that we owe a series of determinations of the solubility of lime in a 10 per cent saccharine solution, at different temperatures, which determinations were given by him in a Report presented in 1876 to the Société Industrielle du Nord.

Lamy endeavoured to determine the amount of CaO which could be dissolved at 30°, 50°, 70°, and 100°, by 100 parts of a decinormal solution of sugar when 1 or 2 per cent of its weight of lime was added. Slaked lime, mixed with the saccharine solutions at the temperature of the experiment, was left for three hours with frequent stirring; the temperature was then raised, and the solution filtered rapidly. Table II. gives the figures found by Lamy for

* In principle, the few reactions we have mentioned in the preceding lines, and which take only a secondary place in this article (as its title shows), are not quite new, and we do not offer them as such. They are mentioned because we have experimented with a large number of monocalcic solutions—all solutions which are included in our tables of the solubility of lime already published, and which we now publish in this article. In this manner we have, independently of our predecessors (Payen), prepared a complete series of sucro-calcic solutions, which, on being heated to 90–95°, become quite solid. It is very simple to prepare saturated sucro-calcic solutions which form a thick paste on being heated to the above-given temperature, while up to the present only a single special receipt had been given for producing this characteristic reaction, which is occasioned, according to some chemists, by the precipitation of a hexacalcic succrate (?), the existence of which is far from having been proved. On the other hand, we read on page 763 of M. von Lippmann's book, entitled "Chemie der Zuckerarten," as follows:—"By dissolving the calcic monosuccrate in 4 parts of water, and boiling the solution obtained, a thick paste is formed which can be turned upside down in the beaker, without any loss." These few remarks will make clear the difference, or the analogy, between our own observations and those of our predecessors in this subject.

TABLE I.—Comparative Results of the Solubility of Calcic Oxide, Hydrate of Calcium, and Milk of Lime in Saccharine Solutions at a Temperature of 15–16°.

Powdered oxide of lime, CaO.			Hydrate of lime, Ca(OH) ₂ .			Milk of lime, Ca(OH) ₂ +aqua.		
Saccharine solution.	Sucro-calcic mixture.	Filtered sucro-calcic solution.	Saccharine solution.	Sucro-calcic mixture.	Filtered sucro-calcic solution.	Saccharine solution.	Sucro-calcic mixture.	Filtered sucro-calcic solution.
Grms. of sugar per 100 c.c. of solution.	Grms. of CaO per 100 c.c. of mixture before filtration.	Grms. CaO in solution per 100 grms. of sugar.	Grms. of sugar per 100 c.c. of solution.	Grms. of CaO per 100 c.c. of mixture before filtration.	Grms. CaO in solution per 100 grms. of sugar.	Grms. of sugar per 100 c.c. of solution.	Grms. of CaO per 100 c.c. of mixture before filtration.	Grms. CaO in solution per 100 grms. of sugar.
13'00	6'37	27'75	13'00	6'00	25'40	—	—	—
11'34	5'52	27'57	11'06	5'55	25'45	11'18	5'40	22'40
10'30	5'10	27'86	10'87	5'07	25'40	10'04	5'00	22'90
9'67	4'70	27'61	9'67	4'50	25'00	—	—	—
8'27	4'00	27'49	8'00	4'12	24'74	8'68	4'27	22'80
7'64	3'55	27'50	8'06	3'72	24'72	7'28	3'80	21'70
6'84	3'32	27'42	6'97	3'32	24'50	6'29	3'20	20'90
5'77	2'60	27'80	5'46	2'60	24'50	5'22	2'72	20'55
4'73	2'17	27'90	4'52	2'25	24'23	4'03	2'18	20'60
3'95	1'70	27'73	3'56	1'63	23'60	—	—	—
3'02	1'23	27'78	2'57	1'30	23'40	—	—	—
2'29	0'92	27'93	1'85	0'88	23'30	2'91	1'54	20'05

TABLE III.—Solubility of Lime, in Three different Forms, in Saccharine Solutions at Temperatures of 80° and 90°.

Dry powdered lime, CaO.			Hydrate of lime, Ca(OH) ₂ .			Milk of lime, Ca(OH) ₂ +aqua.		
Composition of the sucro-calcic solution at the ordinary temperature.		Composition of the same solution heated to 80° and filtered at the same temperature.	Composition of the sucro-calcic solution at the ordinary temperature.		Composition of the same solution heated to 90° and filtered at the same temperature.	Composition of the sucro-calcic solution at the ordinary temperature.		Composition of the same solution heated to 90° and filtered at the same temperature.
Grms. of sugar per 100 c.c. of solution.	Grms. of CaO in solution per 100 grms. of sugar.	Grms. of sugar per 100 c.c. of solution.	Grms. of sugar per 100 c.c. of solution.	Grms. of CaO in solution per 100 grms. of sugar.	Grms. of sugar per 100 c.c. of solution.	Grms. of sugar per 100 c.c. of solution.	Grms. of CaO in solution per 100 grms. of sugar.	Grms. of sugar per 100 c.c. of solution.
13'68	27'8	10'97	12'63	25'5	8'16	—	—	—
10'76	27'6	7'49	11'59	26'0	7'12	11'49	23'1	8'79
6'86	27'7	4'58	9'15	26'0	5'72	8'85	23'1	6'29
4'00	27'4	2'68	8'68	25'5	5'62	5'67	23'1	3'95
4'43	27'8	2'78	—	—	—	—	—	7'65

* Heated to 90° instead of 80°.

the solubility of lime in saccharine solutions at 10 per cent, using 2 grms. of lime per 100 grms. of sugar solution.

TABLE II.—Lamy's Table.

Temperature.	CaO dissolved in 10,000 grms. of decinormal sugar solution.	CaO dissolved in 10,000 grms. of pure water.	Difference, expressing the lime united with the sugar.
100°	15'5	6'0	9'5
70°	23'0	7'9	15'1
50°	53'0	9'6	43'4
30°	120'5	11'7	108'3

We have also made a certain number of experiments for estimating the solubility of lime in saccharine solutions at high temperatures, but we may remark that we do not consider the results yet obtained as completely trustworthy.

We operated in a different manner to that adopted by Lamy. Clear sucro-calcic solutions, saturated with lime at the ordinary temperature, were heated on the water-bath to the temperature mentioned later on. The gelatinous deposit formed was separated from the liquid portion by rapid filtration through a filter kept heated to the same temperature. The liquids separated from the deposits were then cooled to the ordinary temperature, and analysed.

The accompanying Table III. shows the composition of the sucro-calcic solutions before and after heating.

The figures of this table show that, under the conditions of our experiments, the solubility of lime, even at 80° and 90°, is still considerable—much greater, in fact, than that found by Lamy.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 18.

A NEW VOLUMETRIC METHOD FOR THE DETERMINATION OF SILVER.*

By LAUNCELOT W. ANDREWS.

THE chief volumetric methods now in use for silver determinations are those of Gay-Lussac (G. J. Mulder, *Scheikundige verhandelungen*, 1857), Pisani (*Ann. des Mines*, [5], 10-83), Mohr ("Titrimethode," 3rd ed., § 151), and Volhard (*Ann. Chem., Liebigs*, cxc., 1).

Of these, the first, as is well known, is a process of great accuracy, applicable to acid solutions, but is tedious in execution. The second and third are only to be used in perfectly neutral solutions, so that their application is extremely limited, being excluded in those cases where neutralisation produces a precipitate. The method of Volhard is free from these drawbacks, but is seriously interfered with by the presence of substances which impart a yellow or red colour (such as cobalt salts) to the solution, and for those cases in which the amount of silver is extremely small or the solution highly dilute it lacks the sensitiveness of the methods of Gay-Lussac or Pisani, probably because the reaction between sulphocyanates and ferric salts is less delicate as a test for the former than for the latter.

It has therefore appeared to the author to be a matter of some interest to devise a method applicable to acid solutions containing silver along with a variety of other metals, possessing the delicacy characteristic of the starch-

* Presented at the New York Meeting of the American Association for the Advancement of Science, June 28, 1900. From the *American Chemical Journal*, vol. xxiv., No. 3.

iodide reaction. It is the purpose of this paper to present such a method, based upon that of Pisani.

In Pisani's method, a solution of starch iodide is run into the silver solution containing calcium carbonate in suspension.

The exact nature of the reaction which then occurs, and which culminates in decolorisation of the iodide of starch, has not been as yet cleared up in spite of several investigations which have been published on the subject. Silver iodide is precipitated along with oxidised products, silver iodate, hypiodite, or, possibly, silver peroxide in the latter stages of the reaction. Strict proportionality does not exist between the amount of starch iodide consumed and the silver present, although such proportionality is more nearly approached as the solution used is more dilute. The departure from proportionality for solutions between N/100 and N/200 is considerable, as the following experiments show.

In each experiment 25 c.c. of a solution containing 0.7887 m.grm. of silver per c.c. was employed, and the quantity of water added was varied. The results of one set of observations are subjoined.

Expt. No.	Vol. of water added in c.c.	Vol. of Pisani's sol. required.	Character of end reaction.
		C.c.	
41	None	15.70	Obscure, colour dirty.
42	300	18.60	Sharper.
43	None	15.40	As in No. 41.
44	500	20.25	{ Sharp, change from brownish to blue.
45	300	18.70	As in No. 42.

If the effort is made to titrate a silver solution acidified with nitric acid by one of starch iodide, a series of colours may be observed, beginning with yellow and passing through orange, brown, grey, sage-green, &c., without any turning-point being clearly marked.

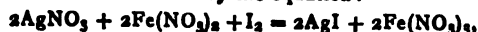
During these changes, the solution exhibits powerfully oxidising properties, more marked than are shown by dilute solutions of either iodine or of iodic acid. The reaction between free iodine and solutions of silver nitrate is now under investigation at this laboratory, and the author hopes to publish definite results before long; in the meantime it will suffice to say that the following general reaction is probable, the equation being written in the sense of the electrolytic dissociation theory:—



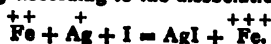
Here the iodine plays a part similar to that which it holds in iodine monochloride and the—*for the present*—hypothetical compound INO_2 is to be regarded as the carrier of the oxidising properties referred to above. It exerts the oxidising action upon organic matter present (as starch), and upon any other reducing substances such as ferrous salts, nitrous acid, or tetrathionates. It undergoes in part hydrolysis into HOI and nitric acid, and in part reacts with the excess of silver nitrate, producing a reddish-brown solution precisely similar in appearance and properties to that obtained by dissolving "silver peroxide" in nitric acid (J. Fischer, *J. Prakt. Chem.*, xxxiii., 237), and which presumably contains silver permittate.

In place of these complicated reactions, a simple one ensues if from the start a sufficient quantity of a feeble reducing agent is present, as ferrous salt or nitrous acid, of such character as not to react with free iodine or starch iodide. In that case a smaller quantity of the solution of iodide of starch will be required than if the titration were conducted according to Pisani in neutral solution, and the end of the reaction is marked by a sharp change from pale yellow to blue.

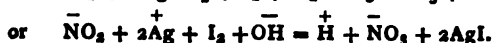
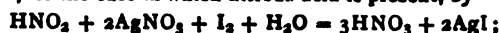
The reaction is shown by the equation:—



or more simply according to the dissociation theory:—



and, for the case in which nitrous acid is present, by—



As might be expected from the respective equations, the reaction in the presence of ferrous salts is practically instantaneous, while that which takes place in the presence of nitrous acid is much slower, especially in very dilute solutions, owing to the slight dissociation of the water and consequent small number of hydroxyl ions that are directly involved in the reaction.

Dilute solutions of ferrous salts, slightly acidified by strong acids, will very slowly decolourise properly prepared starch iodide, with the formation of traces of ferric salts. This action can be checked by the previous addition of ferric salt. Thus, if 2 c.c. of a solution of 40 grms. of crystallised ferrous ammonium sulphate in a litre of water be added to 100 c.c. of water containing 5 c.c. of dilute nitric acid, and a few drops of N/1000 starch iodide then run in to colour the liquid blue, spontaneous bleaching will ensue in about five minutes, whereas, if the iron solution also contain 40 grms. of ferric ammonium sulphate (cryst.) per litre, five to eight hours will elapse before the blue disappears. That there is a condition of equilibrium between ferrous and ferric salts and iodine and iodide, has been shown by Seubert and Dörner (*Ztschr. Anorg. Chem.*, v., 339-411), and by Seubert and Rohrer (*loc. cit.*, vii., 137).

In applying the facts just set forth to the development of a practical volumetric method, the slight solubility of silver sulphate must be borne in mind. If the iron sulphate is added to a too concentrated silver solution, and then titration with starch iodide is undertaken, silver sulphate will be thrown down with silver iodide, and partly enveloped by the latter, making the end-point obscure and the results more or less inaccurate. Some of the experiments described in the following portion of this paper give evidence of this fact and also show the permissible limits of concentration.

Before describing in detail the analytical method, it will be well to say a word regarding the adjustment of apparatus and the preparation of solutions employed.

Apparatus.

All burettes and other pieces of volumetric apparatus employed in the work were carefully calibrated and the readings corrected to true cubic centimetres. The burettes were divided into 1/10 c.c., with divisions sufficiently wide to permit 1/100 c.c. to be estimated with an error of not more than 2/100 c.c. The solution of iodide of starch in the concentrations used is almost perfectly opaque, and permits very exact readings to be taken from the top of the meniscus, which method was uniformly used.* Pipettes and burettes were frequently cleaned with a strongly alkaline solution of potassium permanganate followed by hydrochloric acid, a method to be recommended in preference to that involving the use of sulphuric acid and potassium bichromate commonly employed.

Preparation of Standard N/10 Silver Solution.

Commercial "pure silver 1000 fine" was dissolved in pure nitric acid and water, heated in porcelain, and filtered. An excess of the purest hydrochloric acid was added, and stirring continued until the precipitated chloride had become granular. The precipitate, after being thoroughly washed by decantation, was reduced by digestion on the steam-bath with formaldehyde and ammonia. The silver sponge so obtained was washed with distilled water, compressed and fused in an unglazed porcelain crucible without flux, cast in an ingot mould, treated with fusing caustic

* The use of a float is not to be recommended, according to the author's experience, for either opaque or transparent liquids. Many series of readings made with and without this device, the results being controlled by weighing the liquid delivered, have shown that the float increases instead of diminishing the probable error of a reading, provided the readings are taken in the proper way when no float is used; i. e., with suitable illumination.

potash, and finally cleaned mechanically. The N/10 solution was made up to contain 10.793 grms. (corrected) of this silver per true litre. From this standard a N/100 solution was prepared directly by dilution, and for some of the experiments, other, less pure, silver solutions were standardised by comparison.

Preparation of the Starch Iodide.

The best obtainable commercial maize starch was repeatedly washed with water by decantation, strained through linen, washed with dilute hydrochloric acid, then with pure dilute caustic potash, again with hydrochloric acid, then in turn with water, alcohol, ether, and finally alcohol, and dried at 40°. This purification is not essential, but the starch employed had been purified in this way for the purposes of a correlated research on the properties of starch iodide.

Of the prepared starch, 50 grms. were ground dry with 8 or 9 grms. of re-sublimed iodine until well incorporated, and the powder stirred with 100 c.c. of distilled water, sealed up in large glass tubes, and heated for several hours in boiling water. Instead of heating in sealed tubes, boiling with water in a flask with reflux condenser may be equally well employed, the iodine, which at first sublimes into the condenser, being pushed back into the liquid from time to time. The substance obtained in either way, after moderate dilution, is filtered through a "toughened" (parchmentised) filter. Solution is complete. N/20 standard solutions were made, but N/100 is as far as it is convenient to go in concentration usually. These solutions are as absolutely permanent as any volumetric solutions can be, changing in strength only by evaporation of water. By blowing a current of air through such a solution for a day or two the vapour-pressure of the iodine can be reduced to 0.005 m.m., and even less, as was determined by a method to be published elsewhere.

For all titrations where an iodine solution weaker than N/20 is to be used, as in the titration of alkaline arsenites, antimonous salts, &c., iodide of starch solutions are to be preferred to those prepared in the ordinary way from potassium iodide and iodine, since the former are more permanent, give an even sharper end-reaction, can be more exactly read off in the burette, and avoid the difficulty of preparing a clear, permanent starch solution as indicator or the trouble of always making up a fresh one. The standard of a solution of starch iodide as regards silver is not the same as its standard toward sodium thiosulphate, since only a part (often about two-thirds) of its iodine is in a condition to react with reducing agents, the remainder being in a form analogous to an alkyl iodide, reactive toward silver salts but not toward thiosulphates, and the like.* In some experiments (referred to below) a N/10 solution was prepared (designated "Sol. A") by mixing about 600 c.c. of N/50 starch iodide with about 400 c.c. of ordinary decinormal iodine solution. The mixture was decinormal as regards total iodine. Such solutions can be used when larger amounts of silver are to be titrated, but I believe it to be better in such cases to precipitate the greater part of the silver with a N/10 potassium iodide solution and finish with the N/100 starch iodide, or to use a N/10 potassium bromide, and then, after filtration, to titrate with the starch iodide. Starch iodide reacts with the chloride, bromide, cyanide, or sulphocyanate of silver, so that these salts must always be filtered out before proceeding to the residual titration.

Auxiliary Solutions.

Iron solution C contained 40 grms. ferrous ammonium sulphate and 40 grms. ferric ammonium sulphate with a few c.c. dilute sulphuric acid to the litre. For reasons already given, it is desirable to have solutions of ferrous and ferric salts free from sulphates to use for this method. Experiments were accordingly made with solutions ob-

tained from iron and dilute nitric acid, containing about 28 grms. of iron to the litre, and were successful. Such solutions have the disadvantage of gradually becoming deep brown, in consequence of the formation of basic ferric nitrate, and they contain organic matter derived from the carbon of the iron. This organic matter takes up a little iodine, and therefore decolourises starch iodide. Solutions prepared from strontium nitrate with solutions of ferrous and ferric sulphates are free from the latter objection, but not entirely from the former.

Instead of ferrous solutions, those of nitrous acid may be employed as previously mentioned. They may suitably be prepared by dissolving 30 grms. of sodium nitrite (free from chloride) in 1 litre of dilute nitric acid (13 per cent). Such a solution is designated "Sol. D" in the accompanying tables.

(To be continued).

ARSENIC IN BEER.

By CHARLES ESTCOURT, F.I.C., F.C.S.,
Public Analyst to the City of Manchester.

THERE is no doubt that substances enter into the composition of modern beer which interfere with the usual methods of analysis for the detection of arsenic. This must have forcibly struck every chemist who has analysed beer for the presence of arsenic—as very many have—in beers brewed from arsenical glucose, and failed to detect it. Two of the nostrums used in the brewing of modern ales are finings (which are "cut" with sulphurous acid) and bisulphites of lime and magnesia, &c. These are both used as preservatives, either in washing the barrels, or by adding direct to the beer. If chemists had been acquainted with the presence of these in beer, much less difficulty would have been felt in dealing with arsenical beer. Both these compounds will prevent the discovery of arsenic in beer by the Marsh test, and by the Reinsch also, for unless the beer is boiled some time with acid before putting in the copper the sulphurous acid in the beer will produce the blackening.

Beers brewed from malt, hops, and glucose may be used direct in the Marsh apparatus, and if arsenic is present it will be detected. I have tried the experiment upon beers before racking when no sulphurous had been added. Thus it is not organic matter alone which makes arsenic in beer so difficult of detection, which I proved by taking a beer absolutely free, and adding to it a known quantity of arsenious acid. In the Marsh apparatus I got out practically all I had introduced. To another portion of the same beer with arsenic in such proportion as gave a large deposit in the heated tube, I added a very small quantity of a bisulphite of calcium, and got no deposit of arsenic on the heated tube, only sulphur appearing there. The arsenic was obtained from the flame at the end of the tube when received on porcelain.

The difficulty in connection with the twelve samples of Manchester beer is now explained. I had to do the samples in a day. I did take twelve working hours, and knowing nothing of the presence of sulphite, I found no arsenic. I may say I had only about 11 ounces per sample, and in the small quantities I used for experiments the trace of arsenic was probably lost in the preparation of the beer for the Marsh test. I may premise that I tried a series of experiments with the original beer.

Any test to succeed must deal with not less than 100 c.c., and this should be charred, *only to intumescence*, with acid, then diluted and filtered, and the filtrate evaporated. I did this process in flasks, but found the Medical Officer here was doing the process in porcelain basins, which I now adopt. If one omits to evaporate the filtrate, sulphurous acid may easily be present (carbonaceous matter and sulphuric acid), and spoil the experiment.

City Laboratory,
St. Andrew's Chambers, 20, Albert Square,
Manchester, December 10, 1900.

* Compare Mylius (*Ber. d. Chem. Ges.*, xx., 638). The statement made above is intended to apply to iodide of starch prepared by the aid of heat, as described in the present paper.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 274).

2. Soluble Selenium.

It is familiar to everyone who has carried out the reduction of selenious acid by sulphurous acid, in solution, that when the two liquids are mixed, there is often no precipitation at first; the colour changes from yellow to a more or less deep red, and only after the lapse of a certain time is a deposit of red selenium formed. This makes its appearance then as a red powder, or as a golden or red translucent skin upon the sides of the vessel.

Schulze (1883) has carried out experiments upon these coloured solutions, and his observations, as far as the solubility of the element is concerned, have been confirmed by Muthmann (1887). Schulze's results point to the existence here of a mono- and perhaps a di-selenium tri-thionic acid, $\text{H}_2\text{S}_2\text{SeO}_6$ and $\text{H}_2\text{SSe}_2\text{O}_6$. These acids are instable in the presence of mineral acids, and it is perhaps by this breaking down that the red solutions are formed which afterwards deposit amorphous selenium.

Schulze has observed that the red selenium precipitated from such solutions re-dissolves if an excess of water be added, and that it loses this solubility in a short time, especially if exposed to the light. The solution is stable in the dark, but when allowed to stand in the light, deposits selenium in the form of a red film or skin. By dialysis all foreign substances can be removed. The solution may be boiled without decomposition, but the addition of salts and acids throws out the selenium. Selenium in this form has very strong colouring power; solutions of 1 part in 10,000 parts of water are distinctly reddish.

If we assume that this freshly precipitated selenium forms solutions in the ordinary sense of the word, and loses its solubility on standing, we are led to the conclusion that we have here to do with a distinct form of the element. If, on the other hand, we suppose—what is highly probable—that the acids described by Schulze are colourless, and that the solutions owe their colour to selenium in suspension, then it is quite conceivable that the change in the substance on standing is simply one of aggregation, the extremely small particles deposited from suspension clotting together to larger masses incapable of re-solution. This accords very well with the nature of the red powder and with the familiar observation that at 40° – 50° it clots together to a soft sticky mass.

3. Amorphous Selenium.

Even when freshly precipitated selenium is merely suspended in the liquor, it often remains for hours without settling down. If the vessel be laid aside and the precipitate allowed to come down slowly it forms a soft mass in the bottom of the vessel, resembling in texture and mobility a soft coagulated jelly. When shaken, it does not at first go into powder, but breaks along irregular lines. When dried, it forms an impalpable powder which cannot easily be completely removed from any material with which it has come into contact—the hands, filter-paper, &c.

Hittorf (1851) records that the red powder of selenium when subject to the heat of the sun's rays becomes partially crystalline, but this was probably a heating effect, or he may have been deceived by the change of colour. I have repeated the experiment, and find that there is a rapid change in colour, but the product appears to be still the amorphous form, though much darkened and clotted together.

When gradually warmed, the powder begins to be somewhat adhesive and clots together, and at about 50° forms a soft mass, at the same time darkening in colour; when

this mass is cooled down, it is found to have the brittleness, and, if it has been pressed together, the fracture of the vitreous form. This clotting together on warming is a character of the powders of viscous liquids, as is illustrated by the following experiment:—Some sealing-wax was reduced to as fine a powder as possible in a mortar, then placed in water and warmed. The powder became first of all adhesive, and at 40° – 50° began to clot together. On further raising the temperature, it became quite soft, and after cooling down, it assumed the ordinary brittleness of sealing-wax. The behaviour of amorphous selenium is so exactly similar that it would scarcely have been possible to tell which of the two substances was in hand but for the difference in colour. By placing some of the powder in a pastille press, and striking a few sharp blows upon it with a hammer, a hard black mass is obtained, which has a conchoidal fracture, though less brilliant than that of vitreous selenium prepared in the ordinary way. The red powder is never quite free from dust, which interferes with the experiment. These properties all go to show the identity of the amorphous and vitreous forms. If there be any difference at all between them it is a very slight one, and would not justify their being considered as two distinct modifications. For a discussion of the possibility of such a difference, see the section on specific gravities. Hittorf (1848), Regnault (1851), and others, have remarked upon their identity. The amorphous form of selenium is the one which separates by preference in all cases where selenium is rapidly set free, either by decomposing one of its compounds or by precipitation from solution or vapour. This is in accordance with the rule laid down by Ostwald (*Zeit. Phys. Chem.*, 1897, xxii., 294). The amorphous form is obtained in any of the following ways:—

(1). In the ordinary method of purification by dissolving in potassium cyanide and then re-precipitating with hydrochloric acid.

(2). When selenious acid is reduced by sulphur dioxide. Even if both solutions be previously heated, it is still the amorphous form which comes out. In order to test this further, a boiling solution of selenious acid was added to a boiling solution of potassium sulphite; now at 200° vitreous or amorphous selenium goes over rapidly into the metallic modification; nevertheless, on mixing the above solutions, the selenium makes its appearance first as a red precipitate, and only after the lapse of a certain time does this become black. In place of sulphur dioxide we may use, in this reaction, iron, zinc, hydrogen, phosphorus acid, stannous chloride, ferrous chloride, chromous chloride, &c. Klages (1898) records that there is an emission of light when selenious acid is reduced by hydrogen.

(3). From solutions of selenium in concentrated sulphuric acid, on the addition of water.

(4). By the decomposition of selenious bromide by alcohol (Schneider, 1866).

(5). By sublimation of the element itself, whereby the amorphous form condenses in the cooler parts of the tube, though it is possible also to obtain crystals of the grey form in this way (Bornträger, 1881).

(6). From various compounds of selenium when decomposed, such as the alkaline selenides (Uelsmann, 1860).

(7). By the electrolysis of solutions of selenious acid, or by passing a current through a selenium cell, whereby amorphous selenium makes its appearance at the anode (Bidwell).

When melted selenium is rapidly cooled, the vitreous form is always obtained. This case is not like the above, being merely an instance of supercooling.

Amorphous and vitreous selenium show an increased reactivity compared with the other forms. This is of importance in the purification of the element by the cyanide method, because if selenium be heated in a solution of potassium cyanide, it goes over to the metallic form, and long boiling is then required to bring it into solution;

* From the *Journal of Physical Chemistry*, vol. iv., No. 6.

whereas if the amorphous or powdered vitreous material be placed in a cold cyanide solution, the reaction is complete in a very short time. This fact was observed by Schjellerup (*Ann.*, 1859, cix., 125), but has been lost sight of in most of the more recent work. Its bearing upon analytical methods and upon commercial processes might prove important.

Behaviour of Amorphous Selenium in Solvents.

Mitscherlich (1855) observed that when amorphous selenium is allowed to stand in carbon disulphide for some weeks, it becomes darker in colour and more compact, and changes to a mass of red crystals which are, like the original substance, completely soluble in carbon disulphide, though not readily so. The observation was repeated by Schneider (1866), and later by others. Wishing to repeat the experiment and to find whether the change is influenced by light, I proceeded as follows:—

Experiment 44.—A small quantity of amorphous selenium was placed in carbon disulphide, the tube sealed and allowed to stand in diffused light. After one day the selenium had become very dark in colour, and showed bright crystal faces. On stirring the tube, it was found that this change was only superficial; but upon exposing fresh surfaces, these darkened very rapidly, and in two days the mass had become entirely crystalline in appearance, and had assumed a uniform deep cherry-red colour. The volume of the selenium had also materially diminished. This leads to the conclusion that the change into the crystalline modification goes on much faster than had been supposed, and also that it is in some way affected by light. How far the light effect goes will be better understood from the following experiment:—

Experiment 50.—Amorphous selenium in a tube of carbon disulphide, in the dark. After seven days the mass of selenium had become almost entirely crystalline, but showed no change of colour. After standing for twelve days longer, the colour was a dark cherry-red, much deeper than the original colour, and the entire mass had become crystalline. This change of colour between the two periods may have been due to the exposure of the tube on the first examination, or it may be that the crystals ultimately assume the deeper colour even in the dark. Detailed experiments to trace out the connection between these colour changes were not undertaken, but it is clear that the absence of light, though it retards the darkening of colour, does not prevent the formation of crystals. A later experiment in direct sunlight showed a complete change in one day, the mass becoming uniformly dark in colour and completely crystalline.

After these experiments, I turned to the consideration of the behaviour of amorphous selenium in contact with other liquids.

Experiment 53.—In water. After four days, there was no visible change; after eight days, a perceptible darkening; after fourteen days, this change of colour had become more distinct, but no crystal faces could be observed. During this period of time, the tube had stood at least three afternoons in direct sunlight. After one hundred days, the colour was much darkened, but still red.

Experiment 52.—In absolute alcohol. After four days the powder had become noticeably darker in colour, and this change progressed steadily to the end. After eleven days crystal faces made their appearance, and after fifty days the mass was everywhere crystalline and almost completely black.

Experiment 51.—In benzene. After three days there was a visible change of colour. After four days glittering crystal faces became noticeable, and after six days the mass had assumed a uniform deep maroon, or cherry-red colour, and was everywhere crystalline. During ten days more, there was no change in appearance, and the experiment was then stopped. Experiments 51 and 52 were made under the same conditions as 53 with regard to sunlight.

Experiment 56.—In quinoline. The material becomes black at once, in diffused daylight. Under the microscope the progress of the change may easily be watched. It spreads gradually through the mass under ordinary illumination, and is almost instantaneous in direct sunlight. There is no perceptible shrinking together of the selenium, hence the change is probably not the same as that which is brought about in amorphous selenium by raising the temperature. It remains here in a state of extremely fine division, and when shaken with the quinoline, does not settle again to the bottom for hours. After the first half hour no further change in colour could be observed, although the period of observation extended over forty days. It was impossible to find any signs of crystallinity either with the eye or under the microscope. At the close of the experiment, the selenium was removed from the tube, washed with benzene, and dried. It showed no sign of melting at 215°, but at 225° melted at once. When shaken in carbon disulphide, it remained for a long time suspended in the liquid—much longer than ordinary amorphous selenium—but, on filtering, the solvent came through clear and colourless.

This shows beyond a doubt that the material goes over in quinoline, to the metallic form, and not to the crystalline modification which is obtained, as we have seen, in other liquids. There is here not merely a difference in the rate of change, but a difference in its nature, brought about by the presence of the liquid. It was important then to learn a little more of this change.

A larger quantity of amorphous selenium was placed in quinoline, in diffused light. It remained unchanged for a few moments, then began to darken rapidly, and in fifteen minutes was completely black. After thirty minutes benzene was added, and the material filtered and dried. It did not melt at 200°. The specific gravity was found to be 4.65. A second determination after further washing and drying gave 4.59. The variation is somewhat large, but it must be remembered that only a small quantity of material was used. The results show at least that the change to the metallic form was not yet complete. A larger quantity—about 1 grm.—of amorphous selenium was then placed in quinoline, and left for two days. At the end of that time a specific gravity determination yielded the result 4.86. Hence the change, which is at first so rapid, is only superficial. It is probable that each little particle of the amorphous form becomes covered with a coating of the metallic modification which then protects it from further immediate change.

The question arises, whether the action of quinoline is catalytic, or whether it is an effect of solubility, or in some other way proportional to the amount of that liquid present. To answer this, five experiments were made in liquids composed of benzene and quinoline in the following proportions:—

- | | | |
|------|--------------|---------------------|
| (1). | Pure benzene | |
| (2). | 2 c.c. " | +0.1 c.c. quinoline |
| (3). | 2 " " | +0.2 " " |
| (4). | 2 " " | +0.5 " " |
| (5). | 2 " " | +2.0 " " |

The selenium in No. 3 blackened within a couple of minutes. That in No. 4 was perceptibly slower in changing, but reached the same end state. In No. 3 it was still not entirely blackened after twenty minutes, and that in No. 2 was, after twenty minutes, only very slightly darker than that in No. 1, which had shown no change at all. On the following day, all except No. 1 were completely black. In (4) and (5) the substance remained, after shaking, a long time in suspension. In the others this was much less marked. It appears from these experiments that the velocity of the change is roughly proportional to the concentration of the quinoline.

An experiment to find whether the reaction goes on in the absence of light showed that it did so, although the results were not accurate enough to show whether the velocity suffers a diminution in the dark.

(To be continued).

NOTICES OF BOOKS.

Chemical Technology, or Chemistry in its Applications to Arts and Manufactures. Edited by C. E. GROVES, F.R.S., and W. THORP, B.Sc. With which is incorporated Richardson and Watts's "Chemical Technology." Vol. III. *Gas Lighting.* By CHARLES HUNT. London: J. and A. Churchill. 1909. Pp. 312.

GAS LIGHTING in this country is not yet a century old, and although the electric light is now a powerful rival, inasmuch as it is cleaner, more convenient, and safer than gas, the introduction of the Welsbach and other incandescent burners will no doubt enable it to maintain its important place as an illuminating agent.

The first Gas Company obtained its Act of Incorporation in the year 1810, two years later it was granted a Charter, and was then known for nearly sixty years as the Chartered Gas Company. In 1850 there were thirteen Companies in London; the inconvenience of this was found to be so great that they were eventually induced to amalgamate and form the three which now supply the whole of the metropolitan area from nineteen different gas works.

The process of gas-making is made clear in Chapter II., which is furnished with the plan and section of a modern gas-plant illustrating the complete description given; numerous analyses of gas-coal are quoted in the following chapter. This is followed by details of the process of manufacture and the machinery necessary for the operations.

The various methods for the removal of tar and other by-products are next discussed, together with the necessary washers, scrubbers, purifiers, &c.

Chapter XII. treats of the ammoniacal liquor; full details of an analytical method for determining its value are given, and also descriptions of the more important apparatus devised either for obtaining "*liquor ammonia*" from it, or for converting it into sulphate of ammonia.

Another important class of subsidiary products of gas-making are the cyanogen compounds; these are also fully dealt with.

The storage of gas and its distribution are next considered and the various kinds of gas-holders described; after which we have a chapter on gas-meters, both wet and dry. The wet meter had many disadvantages, and has now been almost entirely superseded by the dry one.

Then follows a chapter on processes for enriching ordinary gas by the use of volatile hydrocarbons, and the Dinsmore process for enrichment by means of coal-tar is also fully described.

The last section of this work is on gas-burners, many different kinds of which are fully described. In conclusion, we have a full account of incandescent gas-burners; these have already effected a revolution in gas-lighting from the brilliancy of the light and the economy arising from the relatively small amount of gas they consume.

The book is profusely illustrated, and is a valuable addition to the literature of the subject.

Action of Light on Solutions of Ferrocyanide of Potassium treated with Peroxide of Hydrogen.—V. A. Kistrakovsky.—A solution of ferrocyanide of potassium exposed to direct sunlight, acquires an alkaline reaction which gradually disappears in darkness owing to the absorption of CO_2 . If we pass a current of air through the ferrocyanide solution in faint diffused light the alkaline reaction is not produced; the voltaic arc causes it to appear slightly. The addition of peroxide of hydrogen greatly increases the sensitiveness. The alkaline solution of ferrocyanide is decolourised when exposed to the light. The author has examined the occurrence of these phenomena, and will shortly publish further experiments.—*Journ. Soc. Phys. Chim. R.*, vol. xxxi., p. 669.

CORRESPONDENCE.

THE
BASES CONTAINED IN SCOTTISH SHALE OIL.

To the Editor of the Chemical News.

SIR,—In a paper on the above subject, read before the Chemical Society on November 15th, Messrs. Garrett and Smythe say:—"But little attention has been paid to those present in shale oil." From this I conclude that they are not acquainted with the work which Mr. G. Carr Robinson and Mr. W. L. Godwin have done in elucidating the composition of the basic shale tars. Their papers will be found in the *Transactions of the Royal Society of Edinburgh*, vols. xxviii. and xxix.

It will be interesting, however, to see whether Messrs. Garrett and Smythe succeed in nitrating these bases, and thus making them available for the manufacture of colouring matters. At present they are burnt under the stills as fuel—I am, &c.,

WM. B. SYME, F.L.C.

Addiewell, December 8, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 21, November 19, 1900.

Direct Combination of Nitrogen with the Metals of the Rare Earth Group.—Camille Matignon.—M. Maquenne described a method (*Comptes Rendus*, cxxi., p. 1147) in which the direct union between nitrogen and the alkaline earth metals was effected. The author utilizes the above method to study qualitatively, in a systematic manner, the action of certain gaseous bodies on those metals which are difficult to procure in the free state or those which have not as yet been isolated. He finds that this method of operation is general and convenient for the qualitative examination of the direct action of certain gaseous or solid bodies on the rare earths. Nitrogen unites directly and rapidly on the following metals:—Thorium, cerium, lanthanum, praseodymium, neodymium, and samarium. Argon does not combine rapidly with the same metals at the temperature of the experiment. Magnesium reduces the oxides of praseodymium, neodymium, and samarium; it being known already, from Winkler's experiments, that this reduction liberates thorium, cerium, and lanthanum from their oxides. The heat of formation of thorium and cerium oxides is higher than that of the other oxides; samarium oxide is apparently the least exothermic. A slight modification of the above method allows of the isolation of the above metals mixed with magnesium. The author is continuing this research in the hope of obtaining the above metals, their nitrides, and their hydrides in a state of purity.

Relation between the Chemical Constitution of the Colouring-matters derived from Triphenylmethane, and the Absorption-spectra of their Aqueous Solutions.—P. Lemoult.—The author experiments on the spectra of various artificial colouring-matters. He comes to the conclusion that the colouring-matters derived from triphenylmethane, which contain, as is generally the case, at least two atoms of nitrogen triply combined to the central carbon, give, when in aqueous solution, an absorption-spectrum containing a band in the luminous red portion of the spectrum when a grm.-molecule is in solution in 1000 litres of water and a thickness of 6 m.m. is used;

the middle of this band occupies an invariable position (wave-length about 6860) for those colouring-matters which have not more than two atoms of tertiary nitrogen, and an invariable but different position (wave-length about 6660) for those which have a third tertiary nitrogen.

Chlorophylline Blue.—M. Tsvett.—In chlorophyll, when defined as the whole of the pigments producing the green colouration of plants, certain yellows and greens have been described. These the author divides into two groups, which may be called the xanthophylline group and the chlorophylline group. The xanthophylline group (carotene, erythrophyll, chrysophyll, &c.) only absorb rays of short period and are not luminescent. The chlorophyllines are fluorescent, and possess, among other things, a characteristic absorption in the red portion of the spectrum. The present paper contains an account of researches made on one of these latter—chlorophylline blue. This substance can be prepared by heating the plant with fine sand, and adding eventually magnesia or calcium carbonate to neutralise the cellular juice. The colouring matter is taken out by benzine, and after undergoing various operations to separate it from the other colouring-matters, an alcoholic solution of the blue pigment is formed. This is slowly evaporated in the cold and crystallises in minute crystals of an inky black appearance.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 12.

The Action of Iodides and Hydriodic Acid on Sulphurous Acid.—A. Berg.—Already inserted in full.

On two Polysulphides of Lead and Copper.—F. Bodroux.—Already noticed.

On a Chlorosulphide of Mercury.—F. Bodroux.—Already noticed.

Crystallised Selenide of Manganese and an Oxyselenide.—M. Fonze-Diacon.—Already inserted in full.

An Arsenide of Nickel.—Albert Granger and Gaston Didier.—Already noticed.

Action of Ethylic Mercaptan on some Diatomic Acetones.—B. Laguet.—Ethylic mercaptan reacts on benzoin in the presence of the dehydrating agents hydrochloric acid or chloride of zinc. It suffices to pass a current of dry hydrochloric acid gas through a mixture of benzoin and mercaptan in excess, or to add finely powdered fused chloride of zinc to the mixture. The transformation into mercaptol is complete in both cases. The solid product obtained is treated and crystallised several times in dilute alcohol; it then appears in the form of white needles fusing at 93–94° without decomposition. Ethylic mercaptan also reacts in a similar manner on benzile, giving white prismatic needles when crystallised in alcohol, and octahedra in acetic acid, both fusing without decomposition at 73–74°, and on acetylacetone, giving a liquid product which decomposes on distillation even under reduced pressure; it has the appearance of an amber-coloured oil, is insoluble in water and has a strong disagreeable odour; its density at 13.5° is 1.008.

Ethylloxalic Anhydride.—L. Bouveault.—Ethylloxalic anhydride is a colourless liquid of a consistency like glycerin, boiling at 135° under 10 c.m. pressure. Its composition is found on analysis to be—C=44.30, H=4.88; calculated for $C_8H_{10}O_7$, C=44.04, H=4.59. It absorbs water with avidity, giving crystals of oxalic acid.

Synthesis of α -Methyl- β -benzoylpropionic (Phenylmethylbutanonic) Acid.—T. Klobb.—Twenty grms. of pyrotartaric anhydride, 40 grms. of $AlCl_3$, and 250 grms. of benzene are heated on the water-bath in a flask fitted with a vertical condenser, for an hour and a half to two hours. The contents of the flask are then treated, while still warm, with water containing a little hydrochloric acid. When the reaction is finished, the upper, almost

colourless, benzenic layer, is separated, filtered, and left to crystallise. The acid, $C_{11}H_{12}O_3$, is deposited in white needles; these are purified by crystallisation in boiling water. The acid, though soluble at 100°, is almost insoluble in cold water. Phenylmethylbutanonic acid melts at 135–136°.

The Acetals of the Phenols.—R. Fosse.—Already noticed.

Action of Chloride of Ethylidene on the Cresols and on Resorcin.—R. Fosse and J. Ettlinger.—Already noticed.

Action of Isocyanate of Phenyl and of Aniline on some γ -Ketonic Acids (II).—T. Klobb.—Already noticed.

The Compounds of Basic with Acid Colouring Materials.—A. Seyewetz.—Already noticed.

Action of Fuming Nitric Acid on Camphene.—L. Bouveault.—According to accepted ideas, the product of the fixation of the molecule of nitric acid on camphene

should be represented by $C_8H_{11} \begin{array}{c} \diagup CH-O-NO_2 \\ | \\ CH_2 \end{array}$, which

makes a nitric ether of a secondary alcohol. It has been shown that the nitric ethers of the secondary alcohols, saponified by potash, give nitrite of potassium and an acetone. In applying this rule to nitrate of camphene, an abundant crystalline precipitate of nitrate of potassium was formed without a trace of nitrite, and the alcoholic solution contained camphene almost exclusively.

On Santalenes and Santalols.—M. Guerbet.—Already noticed.

The presence of Dextrose and Levulose in the Leaves of Beetroots.—L. Lindet.—The author gives several tables of results of the analysis of the sap or juice of the leaves. Samples taken on July 3rd contained 0.89 gm. of dextrose and 0.13 gm. levulose per 100 c.c. of juice; by the 20th, the quantity of dextrose had increased to 2.80 grms. and then decreased again to 1.24 grms. by October 8th. The levulose increased to 0.53 gm. on July 12th, and, after varying between that figure and 0.11 gm., it finally reached 0.37 gm. on October 8th. The proportion of levulose per cent of dextrose varied from 6 to 35.

Essences of Lavender and the Causes of the Variation in their Contents in Ether.—MM. Jeancard and Satis.—Altitude appears to play a secondary part in the proportion of ether present in essences of lavender. The method of distillation is an important point in the richness of the essence in ether. The distillation should not be pushed too far, and should be done as rapidly as possible. The quality of the water used also has an influence; the water should only leave a small residue when evaporated to dryness.

Colorimetric Estimation of Vanadium.—L. Maillard.—Already inserted in full.

Estimation of Lead by Electrolysis in the Sulphate and Chlorides. Application to the Analysis of Lead Glasses and Chromates of Lead.—Ch. Marie.—Already inserted in full.

MISCELLANEOUS.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on the 3rd inst., Sir James Crichton-Browne, Treasurer and Vice-President, in the Chair. The Managers reported that at their Meeting held that day they had elected Dr. Allan Macfadyen Fullerton Professor of Physiology for three years (the appointment dating from January 14, 1901). The following were elected members: Mr. J. Aungier, Mr. E. Barclay, Mr. R. T. Baylis, Mr. H. P. Boulois, Mrs. E. Hills, and Mr. R. Taylor.

Ruthenium and its Compounds. Chlororuthenate of Potassium (II.).—V. Antony and A. Lucchesi.—Chlororuthenate of potassium, RuCl_6K_2 , is obtained by melting together, in a silver crucible, a quantity of ruthenium with 6 parts of KOH, and then adding enough chlorate of potassium to the melted mass to dissolve the whole of the metal. It is further heated to destroy the whole of the chlorate. The mass, which is green when hot and orange-coloured when cold, dissolves in water which has been slightly acidulated with hydrochloric acid, and gives a dark red solution. When evaporated, cold, in vacuo over CaO , it deposits the chlororuthenate in the form of a micro-crystalline red powder, scarcely soluble in water and quite insoluble in the presence of potassic chloride. It is decomposed by hot water. This salt has been analysed by decomposing it, at a dull red heat, in a current of hydrogen; the loss of chlorine was first determined, and then the potassic chloride by lixiviation. Finally, there remained nothing but ruthenium.—*Gazz. Chim. Ital.*, (2), xxix., p. 82.

Estimation of Chromium in Iron and Steel.—E. Dohler.—From 2.5 to 5.0 grms. of the substance are weighed out and dissolved in a matras of 750 c.c. capacity, with 30 c.c. of hydrochloric acid of 1.19 density, and 100 c.c. of water. After complete solution about 750 c.c. of cold water are added, and then an excess of a milk of carbonate of barium. This is left to stand for about twelve hours, keeping the air from it as much as possible by covering the vessel with a watch-glass. Most books on chemistry state that this precipitate will contain the whole of the chromium and the aluminium, as well as small quantities of iron. This, however, is not the case, for the filtrate submitted to fresh treatment with carbonate of barium will, on each successive occasion, give up unmistakable quantities of chromium and a good deal of ferric oxide. It is therefore necessary to precipitate at least twice. The two precipitates are dried, and separated from the filter as much as possible; the filters are incinerated in the same porcelain crucible in which the precipitates are placed, and the whole melted with a mixture composed of 5 grms. of anhydrous sodo-potassic carbonate and 2 grms. of saltpetre. The alkaline chloride formed is taken up with warm water, filtered to remove the iron and the excess of carbonate of barium, and evaporated in the presence of hydrochloric acid to separate the silica; in the filtrate the chromium is separated from the aluminium, and weighed in the state of oxide, Cr_2O_3 .—*Chem. Zeitung*, xxiii., p. 868.

NOTES AND QUERIES.

*. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Pure Zinc.—I should be greatly obliged if any of your readers could tell me of any special purpose, other than German silver or Leclanché rods, for which especially pure zinc (99.90 per cent) would be valuable.—AL. X. B. TUCKER.

MEETINGS FOR THE WEEK.

MONDAY, 17th.—Society of Arts, 8. (Cantor Lectures). "Electric Oscillations and Electric Waves," by Prof. John A. Fleming, M.A., D.Sc., F.R.S.

WEDNESDAY, 19th.—Society of Arts, 8. "The Siege of Ladysmith," by W. T. Maud (Special Artist to the *Graphic*). Microscopical, 8. Demonstration of Lantern Projection in conjunction with the Microscope, by Mr. Barton.

THURSDAY, 20th.—Chemical, 8. "On the Union of Hydrogen and Chlorine," by J. W. Mellor, B.Sc.

ERRATUM.—P. 271, col. 2, line 4 from bottom, for "voltmeter" read "voltameter."

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A BIBLIOGRAPHY OF STEEL WORKS ANALYSIS.

PART VI.

By HARRY BREARLEY.

(Continued from p. 282).

SULPHUR (*continued*).

Sulphur in Fuels.

803. STORER (C. N., xxi., 196).—Coal, pyrites, or free S may be oxidised by heating with $\text{HNO}_3 + \text{KClO}_3$. The BaSO_4 is precipitated from tartaric solutions, and washed with $\text{Am}\bar{\text{A}}$ to remove adherent $\text{Ba}(\text{NO}_3)_2$.
804. BRADBURY (C. N., xxxviii., 147).—Part of the sulphur in coal, *i.e.*, that organically combined, is not oxidised to H_2SO_4 by treatment with acids.
805. DROWN (C. N., xliii., 89).—By separate treatment with NaHO , Br , and HCl , S existing as pyrites or soluble sulphate is dissolved; that organically combined, not. For total S, burn in O, absorb gases in KMnO_4 , and fuse the residue.
806. WALLACE (C. N., xli., 201).—Sulphur *must* exist organically combined when no sulphates are present and not nearly enough iron to form the bisulphide.
807. HELM (Z. I. S. I., 1882, 345).—Gaseous and liquid S compounds are evolved from coal at a temperature below the decomposition of FeS_2 or the volatilisation of S, and therefore part of the sulphur is organically combined.
808. MUCK (Z. I. S. I., 1886, 866).—Evidence that S in coal may be organically combined. The coal containing least sulphur does not necessarily produce the purest coke.
809. BLUM (C. N., lviii., 65).—Wet oxidations remove only such S as is combined with Fe or Ca. Total S obtained by fusion with $\text{KNO}_3 + \text{NaCl} + \text{Na}_2\text{CO}_3$.
810. GRITNER (Z. C. S., lxiv., ii., 89).—Estimates S in coal ash by treating with Br and HCl , precipitating Fe with AmHO , and adding BaCl_2 . This amount subtracted from "total" gives "combustible S."
811. CALVERT (C. N., xxiv., 76).— CaSO_4 should be distinguished from FeS_2 in coal analysis. Boil with H_2O , estimate CaSO_4 in filtrate, oxidise residue with aqua regia, and fuse dried mass with Na_2CO_3 . HNO_3 prevents precipitation of BaSO_4 . In the direct oxidation with aqua regia, sub-sulphate of iron becomes firmly attached to the carbonaceous mass.
812. REICHERT (C. N., xxi., 35).—Ignite charcoal with KNO_3 ; estimate S as usual.
813. A. A. (C. N., xxiii., 30).—Add powdered coke to fused KClO_3 or KNO_3 , and precipitate acidified solution with BaCl_2 .
814. NAKAMURA (C. N., xl., 237).—Heat with mixed alkaline carbonates over a spirit-lamp. Precipitates as usual.
815. ASBOTH (Z. C. S., lxx., ii., 448) and GLASER (Z. C. S., lxxiv., ii., 483).—Fuse with Na_2CO_3 and Na_2O_2 , boil acidified solution with Br , and precipitate as BaSO_4 .
816. ATKINSON (Z. S. C. I., 1886, 154).—As in 780. By heating pyrites with Na_2CO_3 , not more than 96 per cent of the S is retained (see 860).

817. STOLBA (Z. S. C. I., 1888, 524).—Mix with equal amounts of Ag powder and KHCO_3 , heat residue with AmNO_3 , and precipitate as usual.
818. STOCK (C. N., xxx., 211).—Mix coal to paste with CaO , ignite, add AmNO_3 ; re-ignite, dissolve in HCl , and add BaCl_2 . Koninck and Nihoul (Z. C. S., lxxviii., ii., 136) use $\text{CaO} + \text{Ca}(\text{NO}_3)_2$.
819. ESCHKA (C. N., xxxi., 261).—Heat powdered fuel with $\text{Na}_2\text{CO}_3 + \text{MgO}$, re-heat with AmNO_3 , leach, and precipitate S.
820. ROTHÉ (Z. S. C. I., 1891, 952).—The roasting of 819 may be done in porcelain crucibles with less danger of S contamination from the gas.
821. HUNDESHAGEN (C. N., lxvi., 169; and lxxviii., 49).—By using $\text{K}_2\text{CO}_3 + \text{MgO}$ instead of Eschka mixture (819) the combustion is more rapid and the loss as H_2S obviated; this applies to fuels rich in volatile S.
822. HANDY and CAMP (C. N., lxvii., 90).—Hundeshagen's modification is no improvement on the original.
823. PATTINSON (C. N., xxxix., 30).—As 818, omitting AmNO_3 treatment. Determines total S and S in ash.
824. ANTONY and LUCCHESI (Z. I. S. I., 1899, ii., 492).—Heat with $\text{MnO}_2 + \text{KMnO}_4 + \text{Na}_2\text{CO}_3$, acidulate with HNO_3 , and precipitate as BaSO_4 . As accurate as 819, and quicker.
825. PRUNIER (Z. C. S., lviii., 290).—Heat with KMnO_4 , pass gases into KMnO_4 , digest residue with water, add to absorbing KMnO_4 , and treat with HCl and BaCl_2 .
826. NEILSON (C. N., lxiii., 192).—Fuse coke with Na_2CO_3 and MnCO_3 , evaporate with HCl , and precipitate as BaSO_4 . Compares with CaO , MgO , and alkaline fusion processes.
827. MIXTER (C. N., xxvii., 53; and xli., 217).—Burn substances in O and condense H_2SO_4 from the gaseous products. Elaborate apparatus, but process widely applicable. A description of Sauer's process.
828. SAUER (C. N., xxviii., 226).—Burn in O, absorb SO_2 in bromised HCl . See also Marchlewski (Z. C. S., lxiv., ii., 551).
829. BERTHELOT (Z. C. S., lviii., 1462).—Burn with O at 25 atmospheres in a calorimetric bomb; the S is converted to H_2SO_4 .
830. HEMPEL (Z. C. S., lxiv., ii., 187).—Burn the coal electrolytically in a glass bottle, complete oxidation with Br , and precipitate with BaCl_2 .
831. BRUHLE (Z. S. C. I., 1889, 219).—Heat mixture of coal and oxidant in a covered boat, to prevent escape of SO_2 . Results higher than by open fusion.
832. BAILY (Z. S. C. I., 1889, 360).—Imperfections of various methods; prefers 819.

Sulphur in Pyrites, &c.

833. YARDLEY (C. N., xxv., 257).—Pyrites oxidised at high temperatures with HNO_3 do not lose H_2SO_4 , but low temperatures are more favourable both to the decomposition of the ore and the precipitation of the BaSO_4 .
834. LUNGE and BRODENRIG (C. N., xlviii., 285; and lxi., 76).—In opening pyrites with aqua regia, HNO_3 not stronger than 1.42 should be used. Oxidise with Br and HNO_3 , and separate Fe by pouring into AmHO ; the excess must not be boiled off or basic ferric sulphates form.
835. WÖHLER (C. N., xviii., 72).—Pyrites suspended in water is completely dissolved on passing hypochloric acid.
836. JOHNSON (C. N., lxx., 212).—Decompose pyrites with HNO_3 and KClO_3 , reduce Fe in HCl solution with hypophosphite (which also destroys traces of HNO_3), and precipitate with BaCl_2 .
837. STANSBIE (C. N., lxxiv., 189).—Oxidise the sulphide (or even pure S) with HNO_3 and Br . Or heat with CaO ; precipitate S as usual.

838. WESTMORELAND (*J. S. C. I.*, 1887, 84).—Lunge's old and new methods give like results. Only small excess of BaCl_2 should be used and HCl used sparingly in the washing.
839. LUNGE (*J. S. C. I.*, 1887, 96).—Galena does not materially interfere when pyrites is opened up with aqua regia.
840. NOAILLON (*J. C. S.*, lxxii., ii., 595).—Oxidise mineral sulphides with $\text{HNO}_3 + \text{NaClO}_3$, evaporate with HCl , and finish as in 834.
841. TESCHEMACHER and SMITH (*C. N.*, xxiv., 61, &c.).—Precipitated BaSO_4 carries down K , Ba , Cu , Mg , Fe , &c.; it is noticeably soluble in dilute HCl and HNO_3 , and ignited over gas is considerably reduced. Decompose pyrites and titrate HCl solution as in 857.
842. GLENDINNING and EDGAR (*C. N.*, xxiv., 140; and xxvii., 13).—Solubility of BaSO_4 in HCl exaggerated in 841. Gravimetric estimation of S in pyrites a reliable process. Great discrepancies may arise from grinding the mineral in wedgwood or porcelain mortars.
843. GLADDING (*C. N.*, lxx., 181).—Nitrates cause high results; chlorides cause no error. Results low through Fe being precipitated as ferric sulphate. When Fe is precipitated with Am , a little S remains with the $\text{Fe}_2(\text{HO})_6$. Oxidise pyrites with HNO_3 and Br . BaSO_4 is insoluble in Fe_2Cl_6 .
844. LUNGE (*C. N.*, lxxi., 132).—There is no advantage in 843 so far as it professes to be a modification of 834. BaSO_4 is distinctly soluble in acid Fe_2Cl_6 . Process 836 is unworkable.
845. GLADDING (*J. C. S.*, lxx., ii., 622 and 672).—If BaCl_2 be not added in drops it contaminates the BaSO_4 . Lunge contends that it makes no practical difference how the BaCl_2 is added.
846. KÜSTER and THIEL (*J. C. S.*, lxxvi., ii., 247 and 805).—Prevent Fe contaminating BaSO_4 by adding oxalate or tartrate (see 795); or precipitate Fe with AmHO , add BaCl_2 , and then re-dissolve the $\text{Fe}_2(\text{HO})_6$ with HCl .
847. MACAGNO (*C. N.*, xliii., 192).—Digest S ore with a known amount of CS_2 , determine its specific gravity, and read off the amount of S dissolved from tabulated observations.
848. SMITH (*C. N.*, lxii., 206).—Sulphides are oxidised to H_2SO_4 by mixing with molten KHO and passing electric current. BaSO_4 precipitated in a solution free from Fe , &c.
849. REICHARDT (*C. N.*, xlii., 259).—Iron and copper pyrites are oxidised with Br water.
850. LUNGE (*C. N.*, xli., 184).—Dry processes attack both galena and heavy spar, and this partly accounts for the results being higher than by the wet processes.
851. GLASER (*C. N.*, lxx., 179), CLARK (*J. C. S.*, lxxiii., 1080), and ASBOTH (*J. C. S.*, lxx., ii., 71).—Fuse with Na_2O_2 , add HBr , acidify, boil off Br , and add BaCl_2 .
852. DEUTSCH (*C. N.*, xlii., 317) and BOCKMANN (*C. N.*, xlv., 95).—Decompose with $\text{Na}_2\text{CO}_3 + \text{KClO}_3 + \text{NaCl}$; precipitate as usual.
853. CLARK (*J. S. C. I.*, 1885, 329, 573, and 724).—Heat powdered pyrites with $\text{MgO} + \text{NaHO}$, precipitate Pb with CO_2 , acidify, and add BaCl_2 .
854. KELLER and MAAS (*J. C. S.*, lxx., ii., 498).—Copper pyrites and roasted ores are opened up with KHO and Na_2O_2 .
855. JANNASCH (*C. N.*, lxi., 114 and 124).—Heat in bromised air and absorb gases in HCl and tartaric acid. Br may be replaced by HNO_3 . Later, simple ignition in oxygen is preferred (see 827).
856. HINDS (*C. N.*, lxxiii., 285).—Measure the height of a column of liquid containing the BaSO_4 through which a candle flame is just visible. The percentage $\text{H}_2\text{SO}_4 \times \text{depth of column}$ is a constant. AGLOT (*J. C. S.*, lxxii., ii., 431).—Accurate results can be obtained only in alcoholic solutions.
- I.b. Volumetric Estimation.*—
857. WRIGHT (*C. N.*, xvii., 122).—Oxidise pyritic ore with aqua regia, add small excess BaCl_2 , and then Na_2SO_4 until neither Ba nor SO_3 is in solution. Other methods of oxidising the ore; one suitable in case Pb is present.
858. HOLLAND (*C. N.*, xxvii., 15).—A point is reached in 857 when either H_2SO_4 or BaCl_2 will produce a precipitate in the filtered liquid. Pyrites decomposed by fusion in a closed tube carrying a delivery tube.
859. BERRINGER (*C. N.*, lix., 41).—The titration of H_2SO_4 with BaCl_2 is best made in an acetic solution containing soda acetate. A true finishing-point is arrived at (858). The interference of many metals observed.
860. PELOUZE (*C. N.*, v., 45).—Heat powdered pyrites with $\text{Na}_2\text{CO}_3 + \text{KClO}_3 + \text{NaCl}$. The Na_2CO_3 which has not combined (see 816) with the sulphur is titrated. Neither Si , Ba , nor Ca interfere.
861. WATSON (*J. S. C. I.*, 1888, 305).—Heat burnt pyrites with Na_2CO_3 alone, and titrate as in 860. Soluble sulphur is determined by boiling with standard Na_2CO_3 .
862. LUNGE (*J. S. C. I.*, 1893, 292).—Heat as in 861, short of fusion, and extract with NaCl . H_2S may be evolved from FeS when treated with nitrohydrochloric acid; oxidise with HNO_3 and then add HCl (see 771).
863. SCHWARZ (*C. N.*, viii., 207).—Add PbNO_3 , filter off PbSO_4 , and titrate excess Pb with $\text{K}_2\text{Cr}_2\text{O}_7$, using AgNO_3 indicator. Pellet (*C. N.*, xxxiii., 9) used same process, but estimates $\text{K}_2\text{Cr}_2\text{O}_7$ with FeSO_4 and KMnO_4 .
864. CLEMM (*C. N.*, xix., 287), GAWALOWSKI (*C. N.*, lviii., 72), and many others.—To neutral solution add BaCl_2 , then Na_2CO_3 to precipitate excess Ba , and titrate the residue of Na_2CO_3 with H_2SO_4 . NORTH (*C. N.*, lix., 58).—This process is worthless.
865. JEAN (*C. N.*, xxxiv., 272), HAUBST (*C. N.*, xxxv., 27), GROSSMAN (*C. N.*, xli., 114), and CHERIX (*J. C. S.*, lxiv., ii., 89).—Add $\text{Ba}(\text{HO})_2$, precipitate excess with CO_2 , and titrate the alkali (which was combined with the SO_3) with standard acid. Ca and Mg do not interfere.
866. FARNSTEINER (*C. N.*, lxvi., 296).—Add BaCl_2 precipitate excess in faintly ammoniacal solution with K_2CrO_4 , and titrate residual K_2CrO_4 with iodine. Similar processes, varying chiefly in the means of estimating the final K_2CrO_4 , are given by Precht (*C. N.*, xli., 24), Windish (*J. C. S.*, lxviii., ii., 137), and Marboutin (*C. N.*, lxxvi., 232), and others. Marboutin gives a résumé of volumetric processes for estimating S .
867. QUANTIN (*C. N.*, liv., 233).—Add excess of an HCl solution of BaCrO_4 ; SO_3 replaces CrO_3 ; precipitate excess BaCrO_4 with AmHO , and titrate the CrO_3 equivalent to the SO_3 with FeSO_4 . Processes on the same principle are given by Stolle (*J. C. S.*, lxiv., ii., 188), Reuter (*J. C. S.*, lxxvi., ii., 53), Baumann (*J. C. S.*, lxii., 104), and Andrews (*J. S. C. I.*, 1890, 328).
868. ASBOTH (*C. N.*, lxvi., 168).—The HCl solution of BaCrO_4 (867) is gradually decomposed, and is reliable only when freshly made.
869. SOLTZEIN (*J. C. S.*, lx., 115).—Add BaCl_2 , and titrate excess with $\text{K}_2\text{Cr}_2\text{O}_7$, using hæmatoxylin or logwood as indicator.
870. FREUND (*J. C. S.*, lxii., 1377).—Add excess BaHPO_4 , remove BaSO_4 , precipitate $\text{Ba}_3(\text{PO}_4)_2$ with Na_2CO_3 , dissolve precipitate in HCl , add Na_2SO_4 , and titrate H_3PO_4 in the filtered liquid on the principle that, to methyl-orange the acid is mono- and to phenolphthalein it is bibasic.
871. MARCHLEWSKI (*J. S. C. I.*, 1893, 375; and 1894, 283).—Comparative tests and criticism of various

oxidation and evolution methods. Andrew's process (867) can be used in the presence of Mg, Ca, Al, Zn, Fe, Ni, Co, and Ag.

872. COLSON (C. N., xl., 205) and BURTON (J. S. C. I., 1890, 330).—Burn in oxygen, and titrate SO_2 which has been collected in NaHO .

873. NABERY (J. I. S. I., 1896, ii., 456).—As in 872 for coals; titrate excess NaHO with H_2SO_4 . Very little S remains with the ash.

(To be continued).

NOTE ON THE MARSH TEST FOR ARSENIC.

By CHARLES ESTCOURT, F.I.C., F.C.S.,
Public Analyst to the City of Manchester.

SULPHURETTED hydrogen is generated from the purest (arsenic free) granulated zinc obtainable, even when pure diluted hydrochloric acid is used.

Sulphuretted hydrogen is produced in nearly all experiments where sulphuric acid (even diluted) is used with the pure zinc.

Excess of sulphuretted appears to prevent the formation of the brown stain on the heated tube, even where good trace of arsenic is present. Thus, when sulphites are present in beer, scarcely any of the arsenic is obtained.

I use acetate of lead paper in the part of chloride of calcium tube nearest generating flask.

The whole matter deserves further investigation, as these reactions may account for many differences of opinion regarding presence of arsenic.

City Laboratory,
St. Andrew's Chambers, 20, Albert Square,
Manchester, December 14, 1900.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING NOVEMBER 30TH, 1900.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To CHARLES PERRIN, Esq., M.Inst.C.E.,
Water Examiner, Metropolis Water Act, 1871.

London, December 10th, 1900.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 208 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Nov. 1st to Nov. 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 208 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during November has been below the average, though rain fell on twenty days out of the thirty; the actual rainfall was 1.65 inches, the average for the month is 2.42 inches, making a deficiency of 0.77

inch. This brings the total deficit for the year up to 3.22 inches, or 13.6 per cent on the thirty years' average.

Our bacteriological examinations of 541 samples have given the results recorded in the following table; we have also examined 39 other samples, from special stand-pipes, &c., making a total of 580 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	179
New River, filtered (mean of 128 samples) ..	5
Thames, unfiltered (mean of 26 samples) ..	1173
Thames-derived water, from the clear-water wells of eight Thames-derived supplies (mean of 292 samples) ..	17
Ditto ditto highest	236
Ditto ditto lowest	0
River Lea, unfiltered (mean of 26 samples) ..	262
River Lea, from the East London Company's clear-water wells (mean of 43 samples) ..	5

Of the 463 samples of the filtered water supplied to London, examined bacteriologically during the past month, thirteen samples, or 2.8 per cent of the whole number of samples examined, contained more than 100 microbes per c.c. The water supply of the Metropolis continues to maintain the same high standard of purity as we recorded last month.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

A NEW VOLUMETRIC METHOD FOR THE DETERMINATION OF SILVER.*

By LAUNCELOT W. ANDREWS.

(Concluded from page 287).

Experiments on the Influence of Dilution and Degree of Acidity.

In the first series of experiments recorded below, a solution (slightly acid) of silver nitrate containing 0.7887 grm. of silver was used, and an empirical solution of starch iodide, of which 1 c.c. = 1.639 m.grm. of silver designated as "Sol. B."

From these results the following conclusions may be drawn:—

I. Within certain limits the amount of free nitric acid is without appreciable influence.

II. A considerable excess of iron solution is without influence.

III. For increasing dilution a very small correction is called for, which amounts to 0.10 m.grm. of silver for each 100 c.c. of solution. This correction is to be subtracted from the results directly obtained.

IV. If the solution titrated contains much more than 20 m.grms. of silver per 100 c.c. the results are too low in consequence of precipitation of silver sulphate with the iodide. This influence also appears in experiments numbered 58, 59, 62.

The object of the second series, experiments 56 to 62 inclusive, was to show the influence of lead salts upon the titration. It is apparent that there is no influence except when—in consequence of insufficient dilution—the precipitate of lead sulphate carries down silver sulphate with it, as is shown in numbers 58 and 62, and to a less extent in number 59, where the addition of 25 c.c. of water renders the results almost normal. That these errors are really due to the sulphate, and not to the lead, is shown by number 61, in which the same amount of

* Presented at the New York Meeting of the American Association for the Advancement of Science, June 28, 1900. From the *American Chemical Journal*, vol. xxiv., No. 3.

SERIES I.						
Experiment No.	Ag solution. C.c.	H ₂ O added. C.c.	Dilute HNO ₃ added. C.c.	Nitrous acid or iron solution. C.c.	Iodide of starch required. C.c.	Remarks.
46	25	None	—	4 Sol. D	13'90	End reaction sharp.
47	25	250	—	20 Sol. D	13'25	"
48	25	None	1	2 Fe Sol. C	11'85	Ag ₂ SO ₄ precipitated.
49	25	None	5	2 "	11'85	" "
50	25	250	5	2 "	12'20	" "
51	25	250	5	2 "	12'17	" "
52	25	1000	5	2 "	12'65	" "
53	25	1000	20	2 "	12'67	" "
54	25	1000	20	8 "	12'63	" "
55	25	100	5	2 "	12'11	" "

SERIES II.—Two c.c. of Iron Solution (C) added in each Experiment.						
Expt. No.	Ag solution. C.c.	H ₂ O added. C.c.	Dilute HNO ₃ added. C.c.	Pb or Cu solution. C.c.	Iodide of starch required. C.c.	Remarks.
56	25	250	5	10 N/10 Pb(NO ₃) ₂	12'21	
57	25	250	5	10 "	12'18	
58	25	None	5	10 "	11'57	Ag ₂ SO ₄ precipitated with PbSO ₄ .
59	25	25	5	10 "	11'97	" " "
60	25	250	5	10 "	12'20	
61	25	None	5	10 "	12'02	Ferrous nitrate used instead of sulphate.
62	25	None	5	10 "	(v. No. 58) 11'65	
63	25	250	5	10 N/10 Cu(NO ₃) ₂	12'20	
64	25	None	5	10 "	11'88	Ag ₂ SO ₄ precipitated.

SERIES III.—Iodide "Sol. A" (see ante) adjusted to = N/10 Silver Solution. "B" is an Arbitrary Solution of Silver Nitrate.

Expt. No.	Sol. B. C.c.	H ₂ O added. C.c.	HNO ₃ . C.c.	Iron Solution. C.c.	Sol. A required. C.c.	Remarks.
65	25	150	2 conc.	8 Fe sulphates	18'27	
66	25	150	12 "	8 "	18'30	
67	25	150	2 "	2 "	18'25	Very quick titration.
68	25	None	2 "	Fe(NO ₃) ₃ from Fe	18'18	Very slow titration.
69	25	None	2 "	FeSO ₄ + Sr(NO ₃) ₂	18'25	Very slow titration.
70	25	None	10 "	FeSO ₄ + Sr(NO ₃) ₂	18'25	5 c.c. saturated solution NaNH ₄ HPO ₄ .
71	25	150	2 "	8 Fe sulphates	18'27	25 c.c. N/10 Pb(NO ₃) ₂ sol.
72	25	150	4 "	4 Fe nitrate sol.	18'25	
73	25	None	20 dil.	FeSO ₄ + Sr(NO ₃) ₂	18'21	
74	25	150	20 "	FeSO ₄ + Sr(NO ₃) ₂	18'27	
75	25	None	20 "	FeSO ₄ + Sr(NO ₃) ₂	18'21	
76	25	50	—	12 c.c. nitrous acid (Sol. D)	18'15	
77	25	100	—	10 c.c. nitrous acid (Sol. D)	18'22	
78	25	100	—	10 c.c. nitrous acid (Sol. D)	18'25	
79	25	100	—	5 c.c. nitrous acid (Sol. D)	18'23	
80	50	None	—	10 c.c. nitrous acid (Sol. D)	36'36	

lead salt was used, but no sulphate, ferrous and ferric nitrate solution being substituted for it. The titrations in the presence of copper nitrate, numbers 63, 64, show that this salt, as might be expected, is without influence on the result. The solution of lead contained 20.7 grms. of the metal per litre; that of copper 63.3 grms. There was accordingly about five times as much lead present as silver, or about one and a-half times as much copper.

Under Series III. a number of observations have been brought together upon the titration of larger amounts of silver, in more concentrated solution and under varying conditions, with the combined solution of starch, potassium iodide, and iodine. The deductions to be drawn from these data, in addition to those already derived in the foregoing, may be enumerated as follows:—

I. The correction for dilution does not exceed 0.02 c.c. of the N/10 solution for each 100 c.c. additional water.

II. The influence of varying speed of titration is inappreciable.

III. The influence of varying amounts of free acid (above a certain minimum), or of ferrous salt, is inappreciable.

IV. The results obtained by the use of nitrous acid as a deoxidiser are not directly comparable with those obtained when the ferrous and ferric salts are used, being lower when the solution is concentrated and higher (see Experiments 46 and 47) when dilute.

The results with the iron salts are also more constant and the reaction quicker.

V. Phosphates and lead salts are without influence.

Other experiments, not necessary to detail, show that arsenates and antimonates are without influence.

On the basis thus secured it is possible to lay down a general—

Method of Procedure.

The silver solution to be titrated should be acid with nitric acid, but ought not to contain enough of this reagent to oxidise a ferrous salt in the cold; that is, it should contain less than 5 per cent of the acid, HNO₃. It must be free from mercury and from the lower oxides of arsenic and antimony, but may contain sulphurous acid.

Add a ferrous salt in such amount that at least as much iron shall be present as silver, and an equal quantity of a ferric salt. An excess of the latter must be added if the

original solution contained sulphurous acid. If nitrous acid is present, it must first be removed by boiling.

The mixture of ferrous and ferric salts are best kept on hand in the form of sulphates which, just before use, are converted into nitrates by the addition of a moderate excess of strontium or lead nitrate solution, the last step being superfluous when the silver solution contains less than 20 m.grms. silver per 100 c.c.

The titration is to be carried on with constant stirring, and not too rapidly. Under these conditions the end reaction is never in doubt to the extent of even 0.05 c.c., provided that all reagents used are perfectly free from chlorine. A subsequent bleaching of the solution in the course of an hour or two is to be disregarded. A porcelain dish is much more suitable than a beaker for the titration, on account of the uniform white background.

Applications.

The method described is obviously applicable in many ways to residual analyses, as of chlorides, cyanides, &c., and appears especially suitable to the determination of small quantities of these substances, as in water analysis. The author intends to elaborate the process further, and desires to reserve the field for a reasonable time.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 289).

Behaviour of Amorphous Selenium in Solvents (continued).

A NUMBER of experiments were then tried with amorphous selenium in various liquids, the results of which are given below:—

Aniline.—After one-half hour, perceptibly darkened; after two days, completely black.

Thiophene.—After one-half hour, no change; after three hours, crystalline without change of colour. On the following day, the material showing no further change, 0.5 c.c. quinoline was added. After fifteen minutes, the selenium had become black, but there were still glittering crystal faces to be seen. After half an hour, these had all disappeared, and the mass was uniformly black. This experiment demonstrates the power of quinoline to bring about the same change in the red crystals that it causes in amorphous selenium itself.

Pyridine.—After ten minutes, no change; after one hour, completely black.

Toluene.—After five hours, crystalline and darker in colour. No further change after seven days. After thirty days, darker in colour.

Benzonitrile.—After one day, crystalline and darkened. After thirty days more, no further change.

Benzyl Cyanide.—After one day, completely black. After fifty days, no further change.

Benzimidobutyl ester.—After one hour, black; after fifty days, no further change.

Propyl Aldehyde.—After two days, no change; after three days, somewhat darker, but no crystals; after thirty days, very dark and crystalline.

Piperidine.—Black at once.

Amyl Nitrite.—After three days, unchanged; after thirty days, partially crystalline, colour unchanged.

Ethyl Acetate.—After one day, unchanged; after two days, somewhat darkened; after three days, almost black, without crystal faces; after thirty days, completely crystalline.

Isobutyric Acid.—After three days, unchanged; after thirty days, slightly crystalline, no change of colour.

Aqueous Ammonia.—After one hour, completely black; after fifty days, the same.

Acetophenone.—After one day, much darkened and crystalline; after three days almost black, completely crystalline; after thirty days, no further change.

Nitrotoluene.—After one day, crystalline and darkened. No further change after thirty days.

Brom-nitro-benzene (m) in Benzene.—After three hours, crystalline. No further change after thirty days.

p-β-Anisaldoxime in Alcohol.—After one day, much darkened; after two days, completely black, without crystals; after fifty days, the same.

Hydroxylamine Hydrochloride in Water.—After three days, unchanged; after thirty days, somewhat darkened in colour, not crystalline. Potassium hydroxide was then added to liberate the base; this brought about no further change in the selenium.

Disnitrobenzene (m).—After three hours, unchanged; after one day, partially crystalline; after two days, completely so. The change to the crystalline form is always accompanied by a darkening in colour, unless otherwise mentioned.

Dimethylaniline.—After one-half hour, unchanged; after three hours, crystalline; after thirty days no further change.

Nitroso-β naphthol.—After one-half hour, unchanged; after three hours, crystalline; after three days, no further change.

Urea in Alcohol.—After two days, very slightly darkened; after thirty days darkened in colour, not crystalline.

Acetone.—After three hours, darkened; after two days, black, no crystals; after thirty days, black, completely crystalline.

Ammonium Sulphocyanate in Water.—After thirty days, unchanged.

Picric Acid in Alcohol.—After thirty days, unchanged.

Triethylamine.—Begins to blacken at once; after one day, completely black; after fifty days, the same.

Phenylhydrazine.—Begins to blacken after a few minutes; after one day, completely black; after fifty days, black, minutely crystalline.

Benzylamine.—After one day, darkened; after fifty days, crystalline, almost black.

Propylene Bromide.—After one day, completely crystalline, red; after thirty days, much darker in colour.

Diphenylmethane.—After one day, unchanged; after fifty days, deep red, crystalline.

Ethyl Iodide.—After one day, completely crystalline, red.

Hexamethyleneamine in Alcohol.—After one day, nearly black, not crystalline; after fifty days, black.

Acetanilide in Alcohol.—After one day, very slight darkening; after fifty days, considerably darkened; about the same as in alcohol.

Potassium Hydroxide in Water.—After one day, unchanged; after thirty days, considerably darkened, not crystalline.

Ammonium Chloride in Water.—After thirty days, unchanged.

Potassium Ferrocyanide in Water.—After thirty days, unchanged.

Monomethylaniline.—After one day, unchanged; after thirty days, entirely crystalline.

Chloroform.—After one day, unchanged; after thirty days, entirely crystalline.

We see from these results that the substances studied may be divided broadly into three classes:—

1. Those which have little or no action on amorphous selenium.
2. Those which transform it into the crystalline red modification.
3. Those which transform it into the metallic modification.

In the first class we find water, hydroxylamine hydrochloride, hydroxylamine, urea, ammonium sulphocyanate, picric acid, acetanilide, potassium hydroxide, ammonium chloride, potassium ferrocyanide.

* From the *Journal of Physical Chemistry*, vol. iv., No. 6.

In the second, alcohol, benzene, thiophene, toluene, benzonitrile, propyl aldehyde, amyl nitrite, ethyl acetate, isobutyric acid, acetophenone, nitrotoluene, brom-nitrobenzene, dinitrobenzene (*m*), dimethylaniline, nitroso β -naphthol, acetone, propylene bromide, ethyl iodide, monomethylaniline, chloroform, phenylhydrazine, benzylamine, diphenylmethane.

And in the third, quinoline, aniline, pyridine, benzyl cyanide, benzimidobutyl ester, piperidine, triethylamine, hexamethyleneamine, *p*- β anisaldehyde.

The substances which are most active in bringing about the change to the metallic form will be seen to be ring compounds containing nitrogen in the ring; these without exception all show the same behaviour. All the substances which cause this change are nitrogen compounds, but not by any means all nitrogen compounds are capable of causing the change; thus while aniline acts rapidly in transforming amorphous selenium into metallic, both mono- and di-methylaniline change it into the red crystalline form. It is probable that further study in this field would bring out very interesting results. Meanwhile, it looks as if it could not be a question of solubility alone which explains the difference of behaviour of these various liquids.

To determine whether amorphous red selenium would distil over in carbon disulphide to the black form at ordinary temperatures, an experiment was made in a Λ -shaped tube filled with carbon disulphide, some of the amorphous substance being placed at one end and some of the black crystals separated from sulphuric acid in the other; but, although the tube remained for several weeks, no change in the size of the black crystals could be observed. The amorphous material changed in a short time to the crystalline variety, but even this, in virtue of its higher solubility, should have displayed the phenomenon; probably the time allowed was too short.

(To be continued).

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 6th, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

The following certificates were read for the first time:—Messrs. Andrew Charles Aitken, Rio Marina, Isola d'Elba, Italy; Thomas Stewart Barrie, 77, Sinclair Drive, Langside; John Cardew Bedwell, 65, High Street, Colchester; Herbert John Bult, 165, Briarton Hill, S.W.; Gilbert Howard Daniel, 21, Church Walk, Ulverston; Frederick George Donnan, Chemical Laboratory, University College, Gower Street, W.C.; John Alfred Emery, Cinderford, Gloucestershire; Bernard Farmborough Howard, Devon House, Buckhurst Hill, Essex; Nicholas Henry Martin, Ravenswood, Low Fell, Gateshead; Theodore Henry Page, 40, Wilson Road, Camberwell, S.E.; Thomas Slater Price, University College, Sheffield; Lionel Guy Radcliffe, 6, Alma Terrace, Old Trafford, Manchester; William Cecil Ramsden, 13, Effra Road, Rathmines, Dublin; John Talbot, Tunstall House, Harrow; James Waterhouse, Oak Lodge, Court Road, Eltham, Kent; William A. Wayland, 4, Harefield Road, Brockley, S.E.; James Scott Wilson, Bradford Street, Walsall.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—Messrs. Bernard Cracroft Aston; George L. Bennett, M.A.; Hormasji N. Bilimoria, M.A., B.Sc.; Frederick Nisbet Binks; Herbert James Singleton Boyes; Theodore Ridley Burnett, Ph.D.; John Henry Cheeseright; Albert Walter Comber; Alexander Davidson; Arthur Louis William Fechtner; Willie Ludford Freeman, B.A.; William Gasson; George William Gibbings; John

Gibson; Walter Augustus Handcock; William A. Hargreaves, M.A.; George Harker, B.Sc.; Frank C. R. Hemingway; John Brownlie Henderson; Samuel Hewitt; William Henry Hewitt, B.A.; Edmund Foster Hudson, B.A.; Adolf Jappé; Humphrey Owen Jones, B.A., B.Sc.; George Washington Kilner, M.A.; Morris Charles Lamb; George Druce Lander, B.Sc.; William McCall; Hans Meyer, Ph.D.; T. Marginson Nightingale, B.Sc.; Alexander Parry; John Paul; Ernest Vivian Pearce; Henry Ernest Stapleton, B.A.; Arthur Lambert Thornton, B.Sc.; F. Gerhard Wiechmann, Ph.D.; Walter Bourne Woodbridge; Herbert Edwards Wright, M.A.

Of the following papers, those marked * were read:—

*153. "Santalonic Acid." By ALFRED C. CHAPMAN, F.I.C.

The author described the preparation of a crystalline acid by the oxidation of oil of santal wood with a neutral aqueous solution of potassium permanganate. *Santalonic acid* has the formula $C_{13}H_{12}O_2$. It crystallises from dilute alcohol in transparent plates possessing a brilliant pearly lustre, is insoluble in water, but dissolves readily in all the ordinary organic solvents. It melts at 76° , boils without decomposition at 189° (corr.) under a pressure of 28 m.m., and distils with steam.

Its rotation has been determined and gives $[\alpha]_D = +18.05^\circ$.

Many of its salts have been prepared and analysed. The *methyl ester* is an oily liquid boiling at $232-234^\circ$ (35 m.m.), and has a sp. gr. = 1.0132 at $15/15^\circ$. In a 100 m.m. tube at 20° it produces a rotation of $-18^\circ 13'$.

*154. "Ammonium Bromide and the Atomic Weight of Nitrogen." By A. SCOTT.

During a research having for its chief aim the determination of the hydrogen to oxygen ratio by comparing the equivalents of hydrazine, ammonia, and hydroxylamine, the author found that he could not obtain the same equivalent as Stas for ammonium bromide. The value given by Stas is 98.032, whilst that of the author is 97.996, his highest value being 98.003. This would lower the atomic weight of nitrogen from 14.046 to 14.010, a number much nearer that deduced from the relative densities of oxygen and nitrogen (16:14.003).

The bromine used by Stas seems to have contained some impurity, probably platinum, as it turned greyish at 115° , and its greyness increased up to 180° ; he was unable to sublime it even in ammonia without its becoming yellow. The ammonium bromide of the author retains its whiteness at 180° , and has been sublimed in ammonia, in a mixture of hydrogen with ammonia and in a vacuum, giving the same results before and after sublimation. The silver bromide had the same composition as that of Stas (57.445 per cent of silver).

The equivalent for ammonium chloride was also determined, and found to be 53.516, as against 53.532 (Stas) and 53.486 (Marignac).

The silver used was prepared by the reduction of silver nitrate by means of ammonium formate, and was notably better than a sample prepared with the utmost care by the well-known cuprous ammonium sulphite process.

To test the purity of the silver in the severest way, the precipitated silver bromide was reduced by hydrogen, when identical results were obtained with the silver so prepared.

*155. "Relationships of Oxalacetic Acid." By HENRY J. HORSTMAN FENTON, F.R.S., and HUMPHREY OWEN JONES, B.A., B.Sc.

The authors have continued their investigations on the properties and relationships of free oxalacetic acid, obtained by the oxidation of malic acid in presence of ferrous iron.

The behaviour of the acid towards ammonia, aniline, hydrazine, phenylhydrazine hydroxylamine, and urea, was discussed, as well as the relation of the acid to dihydroxymaleic and dihydroxytartaric acids.

It was also shown that the hydrazone of oxalacetic acid readily loses carbon dioxide when heated with water, yielding the hydrazone of pyruvic acid, but that by a sufficient concentration of hydrogen ions this change is prevented, the hydrazone losing water instead, and giving Wislicenus's phenyl-pyrazolone-carboxylic acid; a simple method based upon these changes has been devised for comparing the "strengths" or "affinities" of various acids.

*156. "The Alkaloids of 'Corydalis cava.' The Conversion of Corybulbine into Corydaline." By JAMES J. DOBBIE, D.Sc., M.A., ALEX. LAUDER, B.Sc., and PHOTIOS G. PALIATSEAS.

Corydaline, $C_{18}H_{15}N(OCH_3)_4$, differs from corybulbine, $C_{18}H_{15}NO(OCH_3)_3$, by CH_2 , and contains four methoxyl groups, whilst the latter alkaloid contains only three (*Trans.*, 1895, lxvii., 25). The authors have now proved that corybulbine contains a hydroxyl group and forms a mon-acetyl derivative,—



By treatment with concentrated hydrogen iodide the two alkaloids yield the same phenolic derivative, $C_{18}H_{15}N(OH)_4 \cdot HI$. Corybulbine can be readily converted into corydaline by treatment with equivalent quantities of methyl iodide and potassium hydroxide in methyl alcohol solution. The artificial corydaline agrees in melting-point, solubility in various reagents, and specific rotation with the natural alkaloid. The platinichloride, ethyl sulphate, and hydriodide of the natural and artificial substances have been compared and found to be identical.

*157. "The Inversion of the Optically Active α -Tetrahydro- β -Naphthylamines, prepared by the aid of Dextro- and Lævo-bromocamphorsulphonic Acids." By WILLIAM JACKSON POPE and ALFRED WILLIAM HARVEY.

Dextro- α -tetrahydro- β -naphthylamine dextrobromocamphorsulphonate has been previously prepared from racemic α -tetrahydro- β -naphthylamine hydrochloride (*Proc.*, 1899, xv., 170); it was now shown that on treating the hydrochloride extracted from the mother-liquors with ammonium lævobromocamphorsulphonate, a precipitate of l-tetrahydronaphthylamine l-bromocamphorsulphonate falls, and may readily be obtained in a pure state. The new salt gives $[\alpha]_D = -86.2^\circ$ in solution in absolute alcohol, as against $[\alpha]_D = +86.5^\circ$ for its optical antipodes.

It has been shown that in preparing the benzylidene, benzoyl, and acetyl derivatives of d-tetrahydronaphthylamine, the major portion of the base undergoes optical inversion (*Proc.*, xvi., 74); on liberating the base from its salts by soda, and preparing the hydrochloride, considerable racemisation also occurs, the optically active hydrochlorides being only separable with difficulty from the mixed salts; the enantiomorphously related hydrochlorides gave in aqueous solution $[\alpha]_D = +71.9^\circ$ and -69.7° respectively. The optical inversion seems to occur during the liberation of the base by soda. d-Tetrahydronaphthylamine, d-camphor sulphonate and its enantiomorphously-related isomeride are more easily purified than the corresponding hydrochlorides; these salts crystallise with half a molecule of water, and, after drying, give in aqueous solution $[\alpha]_D = +47.7^\circ$ and -47.4° . The purest specimen of d-tetrahydronaphthylamine, obtained by treating the bromocamphorsulphonate with soda, and distilling under reduced pressure, gave $[\alpha]_D = +37.24^\circ$ in a 100 m.m. tube; this still contained about 30 per cent of the lævo-rotatory base, so that the pure dextro base would have given $[\alpha]_D = +96^\circ$ approximately.

Optical inversion attending the liberation of a base from its salts has not been previously observed, but E. Fischer has shown that leucine and glutamic acid undergo partial racemisation on benzoylation; these substances all contain the group $\begin{matrix} R' \\ R'' \end{matrix} > C < \begin{matrix} H \\ NH_2 \end{matrix}$, and the partial optical inversion is probably due to part of the base being momentarily converted into a substance containing a

group of the type $\begin{matrix} R' \\ R'' \end{matrix} > C: NH_2$ during conversion of an addition compound into one containing triad nitrogen.

*158. "The Alkaloids of 'Hyoscyamus muticus' and of 'Datura Stramonium' Grown in Egypt." By WYNDHAM R. DUNSTAN and HAROLD BROWN.

In a previous paper (*Trans.*, 1899, lxxv., 72), the authors showed that *Hyoscyamus muticus* grown in India contained 0.1 per cent of hyoscyamine which is readily isolated in a pure state. Subsequently, Gadamer (*Archiv der Pharmazie*, 236, ix., 704), in a brief note, recorded the fact that the same plant grown in Egypt contains more than ten times this quantity. Through the kindness of Mr. E. A. Floyer, the authors have been enabled to examine a specimen of the plant from Egypt. They find it to be much richer in hyoscyamine than the sample of Indian growth previously examined. The seeds furnished 0.87 per cent, and the stems and leaves 0.59 per cent. It remains to be determined whether the Indian plant is always poorer in alkaloid or whether it was a peculiarity of the sample examined.

The authors call attention to the Egyptian plant as an important source of the alkaloid hyoscyamine.

Datura Stramonium grown in the Egyptian desert also contains hyoscyamine, unaccompanied by other atropaceous alkaloids, to the extent of 0.35 per cent.

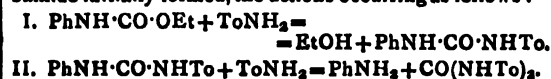
An abundant supply of either plant could be obtained from Egypt.

159. "Interaction of Urethanes and Primary Benzenoid Amines." By AUGUSTUS EDWARD DIXON, M.D.

Manuelli and Comanducci have described (*Gazz.*, 1899, xxix., ii., 142), as phenylparatolylcarbamide, $PhNH \cdot CO \cdot NHTo$, a substance they obtained by heating phenylurethane with paratoluidine; they give its melting-point as $259-260^\circ$, and confirm the formula by a complete analysis. These chemists have apparently overlooked the descriptions of phenylparatolylcarbamide given by Huhn (*Ber.*, 1886, xix., 2408), Goldschmidt and Meissler (*Ber.*, 1890, xxiii., 273), and the author (*Trans.*, 1895, lxvii., 562), who record the melting-point as 211° , 211° , and $212-213^\circ$ respectively; Paal and Vanvolxem (*Ber.*, 1894, xxvii., 2426), give its melting-point as 202° .

It occurred to the author as just possible that Manuelli and Comanducci's compound might be a "labile" form of the symmetrical carbamide, for instance, $PhN:C(OH) \cdot NHTo$; in order to find out if the discrepancy noted above might be thus explained, the interaction of certain urethanes and aromatic bases was investigated.

Phenylurethane and paratoluidine, when heated together, yielded aniline and a crystalline solid; the latter consisted of a main portion melting at $260-261^\circ$, which was proved to be, not phenylparatolyl-, but diparatolylcarbamide, along with a small quantity of phenylparatolylcarbamide melting at $213-214^\circ$. It is suggested that the production of diparatolylcarbamide is due to the expulsion of aniline by toluidine from the phenyltolylcarbamide initially formed, the actions occurring as follows:—



Aniline heated with paratolylurethane gave a solid product which consisted mainly of phenylparatolylcarbamide, m. p. $213-214^\circ$, with only a small quantity of a substance melting at about $256-257^\circ$, probably diparatolylcarbamide.

From orthotoluidine and phenylurethane, aniline always was liberated, the solid product being a mixture, from which were separated (1) vitreous prisms, melting at $249-250^\circ$ (diorthotolylcarbamide), and (2) woolly-looking clumps of needles melting at $203-204^\circ$; these were nearly pure phenylorthotolylcarbamide. The latter compound, when prepared by acting with silver n-trate on the corresponding thiocarbamide, or from phenyl isocyanate and orthotoluidine, melted at $207-208^\circ$.

It seemed therefore not improbable that the *s*-phenyl- α -naphthylcarbamide prepared by Manuelli and Comanducci from phenylurethane and α -naphthylamine, and stated to melt at about 277–278°, might prove to be really di- α -naphthylcarbamide; this interaction was therefore re-examined. Phenyl- α -naphthylcarbamide was prepared in two distinct ways and found to melt at 222–223°. Immediately after melting it re-solidifies, re-melting about 20° higher; the cause of this behaviour has not yet been investigated.

When phenylurethane was heated with a naphthylamine as described, much aniline was formed, together with a solid mixture, which, when boiled with alcohol, left di- α -naphthylcarbamide, which is nearly insoluble, and melts rather indistinctly at 286–287°; from the extract a small quantity of a substance was deposited which crystallised from alcohol in woolly-looking needles. This melted at about 221°, re-solidifying immediately, as described above, and evidently consisted of phenyl- α -naphthylcarbamide.

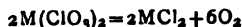
Manuelli and Comanducci also obtained from normal amylamine and phenylurethane a colourless solid, melting at 238°, which they regard as *ab*-amylphenylcarbamide, but which the author thinks is probably carbanilide.

160. "The Decomposition of Chlorates. Part III. Calcium Chlorate and Silver Chlorate." By WILLIAM H. SODEAU, B.Sc.

When calcium chlorate is decomposed slowly about 0.6 per cent of its total chlorine is evolved, whether the pressure be 760 m.m. or 4 m.m., but the proportion exceeds 2 per cent when the decomposition under atmospheric pressure is rendered violent. As the proportion of free chlorine is independent of the pressure of the gaseous products, the calcium chloride in the residue is not produced by the action of chlorine upon oxide first formed.

When silver chlorate is heated to about 350° it explodes with a yellow flash, about 7 per cent of its chlorine accompanying the oxygen. When slowly decomposed the influence of pressure is even greater than in the case of lead chlorate (*Trans.*, 1900, lxxviii., 718). At atmospheric pressure only 0.2 per cent of its chlorine remains free, but 22.6 per cent is obtained at 2½ m.m. pressure, and more than 36 per cent should be obtained if the action between chlorine and silver oxide could be completely eliminated.

It is concluded that oxygen and chlorine are produced during the slow decomposition of either chlorate by two simultaneous independent reactions represented by the equations—



and—



(where M = Ca or Ag). With calcium chlorate the "chloride" decomposition proceeds at about 180 times the rate of the "oxide" decomposition, whilst with silver chlorate the ratio is less than 18:1.

The probability of the "oxide" decomposition being endothermic suggests an explanation of the increase of chlorine in rapid decomposition at high temperature.

Silver chlorate differs from the chlorates of potassium, barium, and calcium in his highly explosive nature, and in the occurrence of an extremely strong secondary action between chlorine and the oxide, but resembles the chlorates of potassium, barium, calcium, and lead, in that no free chlorine is produced by displacement from the chloride.

161. "On Iron Nitride." By G. J. FOWLER, M.Sc.

Nitride of iron was prepared by three different methods: by the action of ammonia on (a) finely divided iron, (b) ferrous chloride and bromide, (c) iron amalgam. Of these methods (a) is the most convenient. The substance was found to correspond to the formula Fe_2N . The temperatures of the formation of iron nitride in ammonia and of its decomposition in hydrogen are identical. These results confirm the conclusions of Stahlschmidt. Heated

in a current of nitrogen, iron nitride begins to decompose at about 600°; it is slightly magnetic; its specific gravity is 6.35.

When oxidised in air or oxygen, only traces of oxides of nitrogen are produced; heated in a current of air, the substance begins to be converted into ferric oxide and nitrogen at about 200°. It takes fire in chlorine ether spontaneously, or when slightly warmed, ferric chloride and nitrogen being formed, but no trace of nitrogen chloride. It is only slowly attacked by bromine, the action probably being due to the presence of hydrobromic acid in the bromine as an impurity. An ethereal solution of iodine has no action on it.

Dilute hydrochloric and sulphuric acids yield the corresponding ferrous and ammonium salts, hydrogen being liberated according to the following equation: $2\text{Fe}_2\text{N} + 6\text{H}_2\text{SO}_4 = 4\text{FeSO}_4 + 2\text{NH}_4\text{HSO}_4 + \text{H}_2$.

The simultaneous action of hydrogen peroxide and sulphuric acid is not distinguishable from that of the acid alone. Nitric acid acts only slowly on the nitride even when strong; the products formed vary with the concentration of the acid.

Gaseous hydrochloric acid begins to attack the nitride at about 220°, and at 350° the reaction becomes rapid, the substance being completely converted into ferrous chloride and ammonium chloride. Gaseous hydrogen sulphide has a precisely similar action at 200°.

Nitric oxide acts similarly to oxygen, and converts the nitride into oxide, the reaction beginning at about 120°, and becoming rapid at 170°. Carbon dioxide oxidises the nitride at about 530°. On heating the nitride in steam at 100°, ammonia is very slowly formed. The nitride heated with sodium and carbon yields sodium cyanide.

It was thought that phenol might act on nitride of iron in the same way as dilute acids, but the two substances do not react.

On heating with ethyl iodide in a sealed tube to 200–230°, gas is produced consisting chiefly of olefines, with a trace of paraffins, but no amines. The reaction may probably be thus represented:—



(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, December 14th, 1900.

Principal O. J. LODGE, President, in the Chair.

By invitation of Prof. Rücker, this meeting was held in the Physical Laboratory of the Royal College of Science.

A paper "On Electric Inertia and the Inertia of Electric Convection" was read by Prof. A. SCHUSTER.

Calculations of self-induction are based on the assumption that the currents which traverse a conductor fill it continuously, the flow being treated as that of an incompressible liquid. The assumption is generally recognised not to hold in the case of electrolytes where electricity is conveyed by a number of irregularly distributed ions. In the immediate neighbourhood of such an ion the magnetic field is many times greater than that calculated on the supposition of continuous distribution, and hence the total magnetic energy is under-estimated. What is universally recognised in the case of electrolytes must also be conceded when the current is conveyed by a gas, and the idea is gaining ground that even in solid conductors the current consists of positive and negative electrons moving with different velocities. It is the object of the paper to calculate the additional terms which become necessary for the evaluation of self-induction, and to discuss the possible cases in which the corrections may effect experimental results. The mathematical investigation shows that it is necessary to add a correcting term containing a quantity which may conveniently be called electric inertia. The

author has calculated the numerical value of this quantity in the case of a solid conductor, and finds it to be about 2×10^{-12} C.G.S. units. It is of the dimensions of a surface. The experiments of Hertz have proved that if electric inertia exists it must be less than 18×10^{-12} . In the case of liquids and gases the electric inertia of the moving ions becomes much more important, and the calculation of self-induction by the ordinary processes gives erroneous results. The introduction of a term representing inertia alters the general equations of electric motion, and the author has applied his modified theory to Leyden jar discharges, the electrodeless discharges of J. J. Thomson, and the electro-magnetic theory of light. In the case of electrodeless discharges in a vacuum globe it is suggested that the absorption of energy may not only be due to the conductivity of the gas, but also to the inertia which it possesses.

A paper "On Magnetic Precession" was then read by the same author.

The most delicate method of investigating the influence of electric inertia is based on the electromotive forces introduced by the motion of conductors carrying electric currents. If electricity behaves like a body possessing inertia, the rotation of a body through which currents pass should affect the flow of these currents in the same manner as the earth's rotation affects the direction of currents of air. If the earth's magnetism is due to electric currents it is interesting to see if the effects of inertia can explain the secular variation. The investigation shows that a magnetic precession of the character of the secular variation would be produced, but that the precession would be very much slower than the variations actually observed. The subject is worked out mathematically, dealing first with the case of currents in a spherical shell, and then extending the result to the case of a solid sphere. The calculated period of a cycle comes out as 7×10^{14} years. If the currents are confined to a thin slice of the earth the time would be reduced to about 14×10^6 years. To produce the actual period of the secular change the current sheet would have to be of molecular dimensions. This suggests the possibility of the phenomenon of secular variation being rather of a molecular than a molar character.

Prof. RÜCKER congratulated the author upon his attempt to solve the problem of terrestrial magnetism, and expressed the hope that further calculation would throw more light upon this difficult subject.

Mr. BLAKESLEY asked if the time of the secular variation would be altered if the interior of the earth were liquid or solid.

The CHAIRMAN observed that the precession was rapid in the case of a thin layer of gas, and mentioned J. J. Thomson's notion that the electrons were thrown off by centrifugal force, and formed a molecular layer. Hertz, in his experiments on electricity, had looked for material inertia besides electromagnetic inertia. In the present theory the distinction disappears, and there is only one inertia, and that electromagnetic.

Prof. AYRTON said if the two forms of inertia were electromagnetic, he would like to know why in detecting the second form it was necessary to associate it with an absorption of energy as in the case of an electrodeless discharge. In the case of ordinary self-induction there is no absorption of energy, and if there is absorption in the second form, and if they are both electromagnetic, he would like to know the difference between the two.

Prof. SCHUSTER, replying to Mr. Blakesley, said that if the interior of the earth were treated as liquid the period of the cycle would be about one hundred times less. In reply to Prof. Ayrton, he said he had only cited one experiment to show that a phenomenon explained by the gas being a good conductor could also be explained by its electric inertia. It was impossible to say in general whether self-induction caused an absorption of energy or not.

Prof. A. W. RÜCKER read a paper "On the Magnetic Field produced by Electric Tramways."

Taking the case of a tramway in which the current flows along a trolley wire from the power house, and returns partly through the rails and partly as earth currents, the author has shown that the vertical disturbing force at any point is due to the currents in the feeders and rails, and that the earth currents affect the horizontal force only. Experiment shows that it is chiefly the vertical force instruments which are affected by the establishment of an electric railway, and since this disturbance is due to the wires and rails it is impossible for an observatory to be protected by rivers or other natural features of the district. A preliminary investigation is based on the assumption that the trolley wires and rails are insulated conductors, and that a fraction of the whole current returns along the rails to the generator. The effect of the railway at a distant point is due to the difference of the current in the trolley wire, and the hypothetical uniform rail current, the effect of which at the point considered is equivalent to the actual rail current, which varies from point to point. It is shown that the disturbance increases with the length of the tramway, and for a tramway of given length the disturbance is a maximum at points on a line perpendicular to and bisecting it. Experiments made at Stockton on the magnitude of the disturbing force gave, with the vertical force instrument, a leakage of 16.3 per cent, and with the horizontal force instrument a leakage of 15.9 per cent; a fairly close agreement. The assumption that the terminals of the line are above and below the average potential of the earth by the same amount respectively, and that the leakage at any point is proportional to the potential difference between the rail and the earth, leads to the ordinary theory of a Fourier bar. This more accurate assumption has been developed and applied to the results obtained at Stockton. The leakage as calculated from the amperes and volts comes out as 20 per cent. The calculation of the disturbing vertical force gives 10.5×10^{-12} C.G.S. units, which is in fair agreement with the value 7×10^{-12} actually observed. In conclusion, it is pointed out that for practical purposes it is sufficient to deal with the average return current through the rails, the formulae for which are quite simple.

Dr. R. T. GLAZEBROOK read some "Notes on the Practical Application of the Theory of Magnetic Disturbances by Earth Currents."

In this paper the author has thrown the extended formula obtained by Prof. Rücker in the previous paper into a workable form, and has tabulated numbers which show at what distances the disturbances are negligible for tramways of different lengths.

Prof. R. THRELFALL exhibited a "Quartz-thread Gravity Balance."

Prof. Threlfall gave a short description of this instrument, which has been described in full elsewhere. He drew attention particularly to its accuracy and portability.

Mr. SIMPSON asked how far the fibre had been calibrated, and if the instrument would be reliable at the freezing-point of mercury.

Dr. GLAZEBROOK asked how far the instrument was suitable for Antarctic expedition work. He drew attention to the difficulty of calibrating a new fibre should one get broken in the field.

Mr. APPLEYARD suggested the use of a bath kept at constant temperature with a thermostat.

Prof. S. P. THOMPSON suggested a Special Meeting to discuss the Physics of the Antarctic Expedition.

Prof. THRELFALL said that there was no difficulty in measuring the relation between temperature and coefficient of stiffness down to very low temperatures. A more difficult matter is the coefficient of temperature of the instrument. Shrinkage of the instrument as a whole affects both the fibre and the spring which supports it. The difficulty of a broken fibre in the field can be got over by taking two or three instruments. Working with a

thermostat is useful in a laboratory, but decreases the portability in exploration work.

Mr. WATSON then exhibited a set of half-seconds pendulums. In these pendulums special attention is paid to the stability of the support. They are covered by a hood from which the air can be exhausted so that the logarithmic decrement is diminished. The motion of the pendulums is shown by rays of light reflected from right-angled prisms attached to them, and the period of oscillation is determined by the method of coincidences. For this purpose an accurate astronomical clock is used, and the observations are made continuously between two time signals. An accuracy of one part in a million is attainable.

In reply to Prof. THRELFALL, Mr. Watson said that the knife-edges were on the pendulums and not on the supports.

The Society then adjourned until January 25th, 1901.

NOTICES OF BOOKS.

Quantitative Chemical Analysis. Adapted for use in the Laboratories of Colleges and Schools. By FRANK CLOWES, D.Sc., Lond., and J. B. COLEMAN, A.R.C.Sc., Dublin. Fifth Edition. London: J. and A. Churchill. 1900. Pp. 592.

THE plan adopted in the previous editions of this book is still adhered to, and endeavour has been made by repeated revisions to eliminate errors, and to include every improvement and useful addition.

An appendix has been added to the present edition, containing descriptions of a modification of the usual method of determining melting-points, of a special apparatus for the rapid filtration and ignition of precipitates by using a perforated crucible lined with asbestos-felt, and an improved form of condenser for use in the distillation of liquids. It also contains descriptions of the following processes:—The determination of nickel by electrolysis, the titration of soluble cyanide by standard iodine solution, the determination of titanium in titaniferous iron ores, and the estimation of nickel and aluminium in steel.

The atomic weights, page 528, used in this volume, are those given for the year 1900, by the Atomic Weights Committee of the German Chemical Society; in this table we note the elements selenium and tellurium. This termination evidently refers to their non-metallic state, but it seems an unnecessary alteration of name.

An Epitome of the Law and Practice connected with Patents for Inventions. By JAMES JOHNSON, Barrister-at-Law, and J. HENRY JOHNSON, Assoc. Inst. C.E. Third Edition, carefully revised. London: Longmans, Green, and Co. New York and Bombay: Stevens and Sons, Limited. 1900. Pp. 164.

THERE are also included in this volume a reprint of the Patents' Acts of 1883, 1885, 1886, and 1888, and the official rules, as well as a summary of foreign and colonial patent laws.

The alterations made from time to time in the law have been carefully shown, and a statement of the procedure to be followed on applying for a patent is given.

Notwithstanding its small size the book will be found to contain a sufficiently adequate outline of the law in its present state.

In consequence of the reduction of fees, and the simplification of procedure since the Act of 1883 came into force, the annual number of applications for patents has considerably increased, and we have no doubt that this book will be of much use in showing in plain language the principal rights, duties, and liabilities of applicants and patentees.

CORRESPONDENCE.

ARSENIC IN BEER.

To the Editor of the Chemical News.

SIR,—I have read Mr. Eastcourt's article as to the interference of calcium bisulphite with Marsh's test with interest. In common with most public analysts I have had lately a large number of samples of beer to test for arsenic. My process has been to boil off alcohol and carbonic acid, to alkalisise with caustic soda, to evolve hydrogen from sheet aluminium, and to pass the gas through a c.c. of silver nitrate solution in a test-tube.

The process will readily detect 0.01 m.grm. of arsenious acid in 100 c.c. of beer. I have proved that the acid sulphites in no way interfere with the test. The only precautions are the obvious ones of control experiments, and the regular slow evolution of the gas.

The samples are put on in the evening, and examined the following morning. An accurate quantitative estimation of arsenic involves, more or less, complete destruction of organic matter, but as a preliminary test the aluminium process is simple, and enables a large number of samples to be tested in a day.—I am, &c.,

A. WYNTER BLYTH.

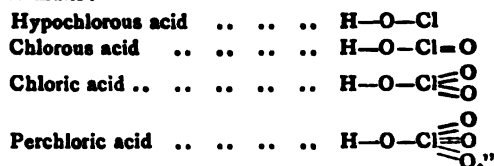
The Town Hall Laboratory,
St. Marylebone, December 15, 1900.

VARIABLE VALENCY.

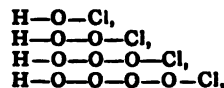
To the Editor of the Chemical News.

SIR,—As a student of elementary chemistry, I have been much puzzled by the following passage in Roscoe's "Lessons" (1888 edition, p. 158):—

"It would appear that chlorine is a monad in hypochlorous acid, a triad in chlorous acid, a pentad in chloric acid, and a heptad in perchloric acid. This is rendered evident by the following graphic formulae:—



Granting that such variable valency is a fact, Roscoe is certainly incorrect in saying that it is "rendered evident" by these formulae. No reason is given for not writing them thus:—



representing chlorine as a monad all through the series.—I am, &c.,

W. F. DUNTON.

26, Tregothnan Road, Stockwell, S.W.,
London, December 5th, 1900.

SIMPLE TEST FOR ARSENIC.

To the Editor of the Chemical News.

SIR,—A want is especially felt at the present time of some quick and ready qualitative test for arsenic. I venture to recommend the following slight modification of Fleitmann's test, which has the advantage of being, like Reinsch's test, applicable in the presence of organic matter, while it is as delicate as, and much simpler than, Marsh's test:—

Place in a test-tube about 6 or 8 c.c. of the liquid to be tested, add about a grm. of solid potassium hydrate, and heat the mixture nearly to boiling. Drop into the solution a bit of aluminium foil, and lay on the mouth of the tube a small piece of filter-paper moistened with solution of silver nitrate. If arsenic is present, hydrogen arsenide will be evolved, and will decompose the silver nitrate, which will turn black owing to formation of metallic silver.

The presence of compounds of sulphur or of phosphorus does not interfere with the above test, since they are retained by the strongly alkaline solution, and no hydrogen sulphide or phosphide is formed.—I am, &c.,

H. G. MADAN.

ANALYSIS OF FERRO-SILICON AND SILICO-SPIEGEL.

To the Editor of the Chemical News.

SIR,—Having read with interest the article on the above subject by Messrs. Ibbotson and Brearley (CHEMICAL NEWS, vol. lxxxii., p. 269), I venture to make a few remarks, as one who has had not an inconsiderable experience in the analysis of these alloys. My experience coincides with that of Hogg, when he states that the silica retains ferric oxide when the alloy is dissolved in aqua regia and evaporated to dryness, &c. But this retention may be entirely obviated by the addition of from 5 to 10 c.c. strong H_2SO_4 when the solution in aqua regia is complete, and then evaporating until the H_2SO_4 fumes; cool, dilute with water, filter, &c. This method is more especially applicable to silico-spiegels. The silica comes out snow-white, and leaves no residue after HF.

As regards ferro-silicon, especially with the higher percentages of silicon, the aqua regia method of solution is very far from perfect. It is difficult, if not impossible, to obtain the silica so clean as would be the case if the following method were adopted:—Take 1 grm. of the finely divided alloy in a dry beaker, add from 5 to 10 c.c. pure bromine, then about 20 c.c. hydrochloric acid (it is a remarkable fact that the bromine should be put on first, then the HCl; also that the beaker is to be dry). Place in a moderately warm spot on the hot plate. Complete solution will be effected in from fifteen to twenty minutes; then add cautiously about 5 c.c. HNO_3 ; copious fumes of bromine come off. When these cease, add H_2SO_4 , 5 to 10 c.c., as in case of silico-spiegels, and evaporate to fuming; cool, dilute filter, &c., as usual. The resulting silica is perfectly pure and leaves no residue after HF. In both cases care is necessary in burning off the residues, as the silica is in a very finely divided condition, and is liable to fly out of the crucible if hurried in the drying and ignition.—I am, &c.,

T. H. BYRON.

Laboratory,
Wigan Coal and Iron Company,
Wigan, December 13, 1900.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 22, November 26, 1900.

The Existence of Nitrides of Neodymium and Praseodymium.—H. Moissan.—Referring to a paper published in the last number of the *Comptes Rendus* on the subject of the nitrides of the rare earths, the author states that, in his paper of October 15th, 1900, he announced the

existence of two nitrides of neodymium and praseodymium prepared by the action of ammonia-gas on the carbides of the above metals at a temperature of 1200°.

Cryoscopic Researches.—Paul Chroustchoff.—From the results of a large number of researches on the subject of the lowering of the freezing-point of water containing in solution salts and organic substances, the author finds many contradictions, and so undertakes the same research, substituting for the mercury thermometer Callendar and Griffith's electric thermometer, which was slightly modified to meet the required conditions. From the numbers obtained he concludes that—(1) There exist solutions in which the coefficient of the lowering of the freezing-point does not vary with dilution, as NaCl, within the limits of the experiment, but that of KBr diminishes slightly with dilution. (2) For other solutions, this coefficient increases in a very marked manner in certain cases, as K_2SO_4 solution, or in a very slight degree as cane-sugar solution. (3) The results agree, at least in their general character, with those of M. Ponsot.

New Method of Estimating Arsenic.—O. Ducru.—In a weak ammoniacal solution, rich in ammonia salts, arsenic acid can be completely precipitated by cobalt salts. This reaction can be employed in estimating arsenic. The conditions necessary are very simple: the cobalt must be in decided excess, about one and a-half times the theoretical quantity, and the solution should be slightly ammoniacal. If it contains in the free state 1.5 per cent of its volume of 20 per cent ammonia, the conditions for the formation of mono-ammonic arsenate of cobalt are fulfilled. Practically, to allow for loss during heating, about 3 per cent should be taken. It is also necessary to charge the liquid with ammonium chlorohydrate, about 100 grms. per litre, though in certain cases it is preferable to use acetate.

A General Method of Separating the Metals which accompany Platinum.—E. Leidié.—The commercial extraction of platinum and iridium from their minerals leaves a residue which contains a number of rare metals, such as osmium, ruthenium, palladium, and rhodium. The author devises a method of separating these metals from one another, based on the properties of their nitrides.

Direct Combination of Hydrogen with the Metals of the Rare Earth Group.—Camille Matignon.—The author finds that neodymium, praseodymium, and samarium combine directly and rapidly with hydrogen. Thorium, cerium, and lanthanum are already known to absorb hydrogen with the same rapidity. All the hydrides thus formed are dissociable. The method which served to establish the above facts at the same time allows of the measurement of heterogeneous dissociation pressures of these hydrides in a very simple manner.

Some Chlorobromides of Thallium.—V. Thomas.—The author prepared a number of mixed salts either by the action of variable quantities of bromine on thallous chloride or by the action of bromine on the products of decomposition of chlorobromides under various conditions. The first salt examined was the chlorobromide $Th_2Cl_2Br_4$, which was prepared in a well crystalline form by treating thallous chloride with excess of bromine, and evaporating the solution in a vacuum over sulphuric acid. The crystals thus formed are yellow, and apparently undergo a change on exposure to air. They are decomposed by water.

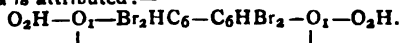
Cadmium Selenide.—M. Fonze-Diacon.—The author prepares a crystallised cadmium selenide, $SeCd$, which is rhombohedral and isomorphous with zinc selenide obtained under the same conditions. He also shows the existence of cadmium chloro-, bromo-, and iodo-selenides. The iodoselenide, which is yellow in the presence of water, becomes bright orange-red on desiccation, and is very stable.

Detection of Metals contained in very small quantities in Mineral Waters.—F. Garrigou.—The results obtained up to the present time during the ordinary

analyses of mineral waters on 1 or 2 litres of water, give no idea of the number and the quantity of metals which are contained in these waters, transported from the deep soils. If 10 to 20 litres are used and the most delicate tests are applied, the presence of various metallic radicles can be established which ordinary analysis fails to identify. The author thus examined water from Eaux-Bonnes, Bagnères-de-Bigorre, Bourboule, and Barèges, and confirmed his old analyses made on thousands of litres. He intends to show that this method of procedure allows also of the separation of the various organic matters from water (acids and alkaloid).

Nitration of Bi-substituted Derivatives of Benzene.—Ch. Cléz.—Noelting and others have fixed certain rules relating to the nitration of mono-substituted derivatives of benzene, and from a study of these the author formulates a series of rules for bi-substituted benzene derivatives, which he enunciates as follows:—(1) The bi-substituted benzene derivatives enclose the basic group NH_2 (acetyl) or NR_2 . (2) The bi-substituted benzene derivative is a phenol (OH being feebly acid). (3) The bi-substituted benzene derivative contains a neutral group. Cl or CH_3 ; and (4) it contains the two acid groups COOH and NO_2 .

Action of Nitric Acid on Tribromated Gaïacol.—H. Cousin.—When tribromated gaïacol is treated with nitric acid, two molecules of gaïacol lose each an atom of bromine, and thus form a derivative resulting from the joining of the two benzenic radicles. At the same time the two molecules of gaïacol are saponified and transformed into phenolic functions. Nitric acid then acts as an oxidiser, and removes H_2 from two phenolic oxyhydrils OH; thus a quinone is formed to which the following formula is attributed:—



Bulletin de la Société Chimique de Paris.
Series 3, Vol. xxiii., No. 13.

The Lower Oxides of Phosphorus.—M. Besson.—A controversial paper, traversing the statements recently made (*Lieb. Ann. Chem.*, cccx., 45) by MM. Michaelis and Pitzch.

Wet Reaction by which Mercuric and Mercurous Iodide can be obtained directly in the Crystalline Form.—F. Bodroux.—Already noticed.

Partial Synthesis of Left-handed Erythrite.—L. Maquenne.—This substance is obtained by the hydrolysis of erythrose acetamide by means of boiling dilute sulphuric acid; it is then neutralised with soda, evaporated in vacuo, and shaken up with 3 per cent sodium amalgam, keeping the liquid slightly acid to prevent the isomerising action of an excess of alkali. After about an hour the solution is carefully neutralised, concentrated on the water-bath until crystallisation commences, taken up with alcohol to separate the insoluble sulphates of sodium and ammonium, evaporated down again, and finally taken up with four or five times its volume of absolute alcohol; it is then saturated with hydrochloric acid gas, and finally treated with a large excess of benzoic aldehyde, which immediately precipitates the erythrite in the state of insoluble acetal. This latter is collected after twenty-four hours, washed with alcohol at 50° , and hydrolysed by boiling for about half-an-hour with ten times its weight of alcohol containing three or four hundredths of sulphuric acid. When the solution is complete, the alcohol and the benzoic aldehyde are driven off with a current of steam; it is then neutralised with chalk, evaporated on the water-bath, and taken up with boiling absolute alcohol. This last solution soon deposits crystals of pure *l*-erythrite.

Researches on Symmetric Diphenylcarbazide. Organo-Metallic Compounds of Diphenylcarbazone.—P. Cazeneuve.—Already noticed.

Some Compounds of Diantipyrine-methane (Formopyrine).—G. Patein.—Ten grms. of iodine and 10 grms. of formopyrine are each dissolved in 150 to 200 c.c. of alcohol at 95° ; the solution of iodine is poured drop by drop into the solution of formopyrine while constantly stirring; fine needles soon begin to appear in the liquid, and they increase with every addition of iodine; after standing for some hours in a cool place the crystals are collected on a filter-paper, washed with alcohol at 95° , and dried in the air. They have the appearance and colour of iodine, but are stable at a fairly high temperature, and give off no vapour. Analysis shows that this body contains 1 molecule of formopyrine combined with 4 atoms of iodine. Formopyrine will not combine directly with pyrocatechin, but if treated together at 100° , in the presence of sulphuric acid, a crystalline body is obtained. In the same way formopyrine and hydroquinone give a colourless mass of crystalline needles, fusible at $218-220^\circ$. Formopyrine and resorcinol also give colourless crystals, fusing at 220° .

MISCELLANEOUS.

Royal Institution.—On Thursday next, December 27, at three o'clock, Sir Robert S. Ball, F.R.S., will deliver the first of the Christmas Course of Lectures, specially adapted to young people, at the Royal Institution, his subject being "Great Chapters from the Book of Nature." The remaining Lectures will be delivered on December 29, and January 1, 3, 5, 8, 1901.

Qualitative Separation of Antimony and Tin.—G. Bornemann.—When we add a quantity of soda-lye to a solution of chloride of antimony, until the precipitate formed is re-dissolved, and then treat this solution with a sufficient quantity of phosphate of sodium and boil, there is no precipitate formed as long as the liquid is warm. A solution of stannic chloride, subjected to the same treatment, loses nearly the whole of its tin in the form of a dense, white precipitate. But on treating a mixture of chloride of antimony and chloride of tin in the same manner, the precipitate of stannic phosphate always carries down a certain amount of antimony. However, the filtrate is free from tin, and sulphuretted hydrogen only causes an orange precipitate, or cloudiness of sulphide of antimony. These facts have enabled me to devise the following process:—The liquid to be examined should contain all its tin in the form of stannic chloride. We then add a drop of phenolphthalein, or litmus, and enough soda to obtain a clear solution with a strongly alkaline reaction. The solution thus obtained is treated with a sufficient excess of a solution of phosphate of sodium, and the whole made slightly acid with nitric acid. It is then boiled, and left to stand for some time. If no precipitate is formed it is diluted and heated over again. The continued absence of a precipitate shows the absence of tin. The phosphate of tin is thrown on a filter, and the filtrate treated with sulphuretted hydrogen.—*Zeitschrift für Angewandte Chemie*, xii., p. 635.

MEETINGS FOR THE WEEK.

THURSDAY, 27th.
SATURDAY, 29th.

Royal Institution, 3. (Christmas Lectures to Young People). "On Great Chapters from the Book of Nature," by Sir Robert Stawell Ball, D.Sc., LL.D., F.R.S.

TO CORRESPONDENTS.

W. L. Watson.—See "Coal-tar and Ammonia," by Dr. George Lange (third edition), published by Gurney and Jackson, 1, Paternoster Row.

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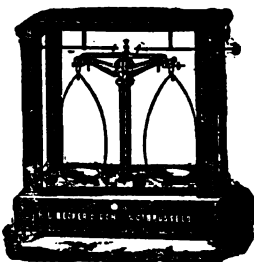
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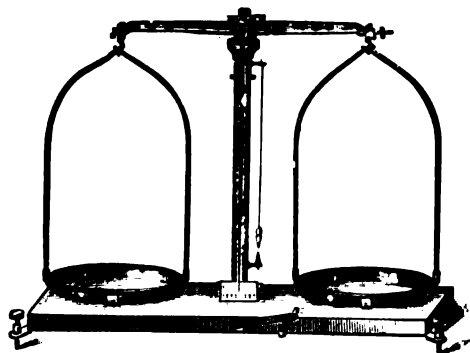
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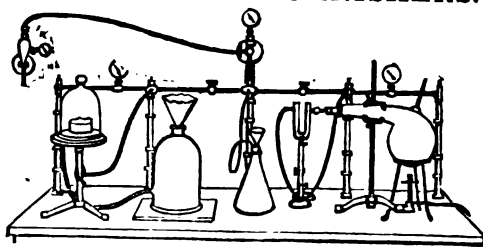
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VOL. LXXXII., No. 2144.

THE DETECTION OF ARSENIC.

By ALFRED H. ALLEN.

THE recent correspondence in the papers, emanating chiefly from chemists who have been concerned in the deplorable epidemic of arsenical poisoning in Lancashire, has led to some interesting disclosures. Especially curious are the statements made respecting the delicacy of Marsh's test for arsenic, and its suitability for examining beer for that element.

Mr. William Kirkby states that it is doubtful whether Marsh's test, under the most favourable conditions, will indicate less than one grain of arsenic in 100,000 parts, or say about three quarters of a grain per gallon of beer. This astounding experience may be contrasted with the statement of Mr. Frank Scudder—apparently rather on the dubious authority of the text-books—as a result of his personal experience, that a mirror on a porcelain basin, in which you can see the reflection of your face, can be obtained from $\frac{1}{2}$ th of a gallon of a liquid containing one part of arsenious oxide in 700,000. But Mr. Scudder does not in practice employ these quantities, preferring to operate on not less than 1 litre of the beer.

Mr. William Thomson finds that, working on 150 c.c. of the beer, an amount of arsenic equal to one-hundredth of a grain of As_2O_3 can be detected with certainty by Marsh's test. He attributes the difficulty many analysts experience in detecting arsenic in beer to the imperfect or unsuitable methods they adopt to destroy the organic constituents of the liquid.

Mr. Chas. Estcourt has done good service by reminding analysts of the frequent presence of sulphites in beer, and of the fact that such beer will not give an arsenical mirror with Marsh's test. It is well known that sulphites evolve sulphuretted hydrogen when treated with zinc and dilute acid, and if this is produced in sufficient amount it may cause complete conversion of the arsenic into sulphide in the evolution-flask, and so remove it from the sphere of action. But I have in mind a case in which the material (burnt pyrites), when examined by Marsh's test, gave a yellow deposit of arsenious sulphide just beyond the heated part of the tube. At any rate, it is clear that sulphites interfere with the employment of Marsh's test, and should be previously removed. Mr. Estcourt effects this by boiling the beer for some time with acid before adding the metal. This appears to me an uncertain and tedious method, and I effect the immediate destruction of any sulphites by adding a little bromine-water, or potassium permanganate, to the acidulated beer. I prefer bromine-water, as it does not act on the organic matter so readily as permanganate, and the excess can be readily removed by a few minutes' boiling. Whichever plan be adopted, the arsenic is oxidised to the arsenic state, and some time elapses before the liquid will evolve arsenical hydrogen in Marsh's apparatus. This inconvenience I avoid by adding a few drops of a solution of cuprous chloride in hydrochloric acid to the contents of the evolution-flask. This reagent instantly reduces the arsenic to the arsenious state, and arsenical hydrogen is evolved almost immediately. The use of cuprous chloride has the additional advantage that it results in the formation of a copper-zinc couple, and the evolution of hydrogen is much facilitated.

A working difficulty in the examination of beer by Marsh's test is met with in the persistent frothing of the liquid; this may be in a great measure overcome by pre-

viously boiling the beer till it is reduced to about half its original bulk. Even then a capacious flask is necessary. Oxidation of the organic matter also mitigates the difficulty.

Mr. Kirkby has described his method of applying Gutzeit's well-known mercuric chloride test for arsenic. It is undoubtedly very delicate, and is used by Mr. Kirkby on 3 c.c. (!) of the beer. But the test does not appear to present any other advantages, and a yellow stain on a piece of filter-paper does not appeal to my mind in the same way as an arsenical mirror, or the formation of crystals of characteristic form. Messrs. Paul and Cownley fully endorse this view, and consider that to obtain a satisfactory result with the mercuric chloride test is out of the question.

I have recently had a considerable number of samples of beer to examine for arsenic, and find Reinsch's test to be the most satisfactory. I purify the hydrochloric acid employed by distilling off about one-tenth; this fraction containing the minute trace of arsenic originally present. I operate, as a rule, on 100 c.c. of the beer, and as a preliminary treatment to eliminate sulphites, add hydrochloric acid and a little bromine-water, and boil the liquid for a few minutes. To obviate the difficulty caused by the fact that arsenic acid only responds to Reinsch's test after prolonged boiling, and in presence of much acid, a little solution of cuprous chloride in hydrochloric acid is next added, which reduces the arsenic to the arsenious condition. On now introducing about one square centimetre of copper-foil, and boiling, any arsenic is promptly deposited on the copper. The boiling is continued for thirty minutes, any water lost by evaporation being replaced. If the copper has undergone darkening it is dried in the water-oven, cut into strips, and heated in a narrow tube, when a characteristic deposit of arsenious oxide, in the form of microscopic octahedra or tetrahedra, will be obtained if the deposit on the copper was due to arsenic. Unless these crystals can be obtained I am not satisfied that arsenic is present. In doubtful cases, a better definition of the crystals can be obtained by filling the tube with water, which acts only very slowly on crystallised arsenious oxide, and, in my opinion, is preferable to alcohol.

This process has the advantage that the arsenic is actually seen and identified as such. Several such deposits can be united, and the arsenic again deposited on copper or subjected to Marsh's test.

I leave the question of the determination of arsenic in beer for a future communication.

Since the foregoing notes were written the Expert Committee appointed by the Manchester Brewers' Central Association have issued their report. They strongly advocate Reinsch's test, but make no reference to the importance of ensuring that the arsenic is in the arsenious condition. The omission has called from Mr. J. A. Wanklyn a reminder that an "eminent German chemist" (name not stated), in 1861, made an elaborate investigation which showed that 20 grains of arsenic "in one of its commonest forms" (name not stated), dissolved in a gallon of liquid would remain undetected by the method recommended by the experts. One detail in the expert's process is very valuable, and that is the direction to warm the upper part of the sublimation-tube before heating the copper. This precaution results in the deposition of much larger crystals of arsenious oxide than are otherwise obtained.

In the CHEMICAL NEWS of December 21st Messrs. A. Wynter Blyth and H. G. Madan independently recommend the treatment of beer with caustic alkali and aluminium, and utilise the reaction of the evolved hydrogen with silver nitrate as a proof of the presence of arsenic. The method is very simple, but the formation of a black stain or precipitate does not appear so convincing as the formation of crystals.

Sheffield, December 18, 1900.

A BIBLIOGRAPHY OF STEEL WORKS
ANALYSIS.

PART VI.

By HARRY BREARLEY.

(Concluded from p. 295).

SULPHUR (*continued*).

II. EVOLUTION AS SULPHURETTED HYDROGEN.

II.a. Gravimetric Estimation.—

874. ABEL (*C. N.*, vi., 124).—Dissolve Fe in strong HCl, absorb H_2S in PbA_2 , and convert PbS into and weigh as $PbSO_4$.
875. FORBES (*C. N.*, xvi., 105).—Digest with cold HCl, pass H_2S into ammoniacal $ZnCl_2$, boil with HNO_3 , and precipitate as $BaSO_4$.
876. HAMILTON (*C. N.*, xxi., 147).—Absorb H_2S in KHO, oxidise with chlorine, and add $BaCl_2$. Oxidise residue with aqua regia, and collect the small amount of precipitated $BaSO_4$.
877. REINHARDT (*J. I. S. I.*, 1885, 650).—Absorb in KHO, oxidise with Br and HCl; or absorb in alkaline PbO , and convert PbS to $PbSO_4$. (*J. C. S.*, lviii., 1464).—NaHO is used containing a known amount of H_2SO_4 in order to assist the collection of the H_2SO_4 from the iron.
878. DROWN (*C. N.*, xxix., 201).—Absorb H_2S in permanganate. decompose the $KMnO_4$ with HCl, and add $BaCl_2$.
879. PETER (*J. I. S. I.*, 1885, 618).—As 878; using concentrated $KMnO_4$, and applies the process to ferro-silicon.
880. SCHNEIDER (*J. I. S. I.*, 1893, ii., 532).—Permanganate the best absorbent for H_2S ; neither the residue nor the solution of the metal contains S. The treatment of ferri-ferrous $BaSO_4$ with HCl only makes bad results worse.
881. AUCHY (*J. I. S. I.*, 1896, ii., 445).—Absorb H_2S in alkaline $KMnO_4$, decompose with HCl and oxalic acid, and precipitate as $BaSO_4$. Determine S in the evolution flask residue.
882. KONINCK (*C. N.*, xxiii., 204).—Collect in $AgNO_3$, digest Ag_2S with Br, remove $AgBr$, and precipitate H_2SO_4 as usual; or absorb in neutral Cd salt and titrate liberated acid with KHO. Later (*C. N.*, lviii., 208), absorbs H_2S in $Hg(CN)_2$ and $AmCl$, so as to avoid washing the $AgBr$.
883. MORRELL (*C. N.*, xxviii., 229).—Pass evolved H_2S into ammoniacal Cd solution; precipitated sulphide dried and weighed.
884. LUTSCHER (*J. I. S. I.*, 1888, i., 379).—Absorb H_2S in alkaline PbO , boil, and settle the PbS by whirling the graduated tube in a centrifugal machine. Accurate to 0.003 per cent.
885. SCHULTE (*C. N.*, lxxv., 47).—Pass H_2S through heated tube into CdA, add $CuSO_4$, filter off CuS , and ignite to CuO . Both P and As interfere if the solution is passed directly into Cu solutions.
886. CRAIG (*C. N.*, xlv., 199 and 272).—Absorb in ammoniacal H_2O_2 , acidify, and add $BaCl_2$. All the S is evolved as H_2S , even from cupriferos and white irons (see 891 and 904).
887. MEINECKE (*J. I. S. I.*, 1888, ii., 333).—As 886; in a current of H, and absorb in $H_2O_2 + NaHO$.
888. REIS and WIGGERT (*J. I. S. I.*, 1891, ii., 325).—Evolve in current of CO_2 , absorb in $AmHO + H_2O_2$, and precipitate with $Ba(NO_3)_2$ from an exactly neutral solution. The precipitate is easily filtered.
889. REIS (*J. I. S. I.*, 1895, i., 509).—Use HCl of 1.09 sp. gr.; boil to thick fluid, and absorb H_2S in $AmHO + H_2O_2$. H_2O_2 may contain H_2SO_4 or fluo-silicic acid. CO_2 must not be used to carry H_2S forward. By boiling H_2O_2 solution, S may be oxidised beyond H_2SO_4 , difficult to precipitate with $BaCl_2$. All these points are guarded against.

890. SPULLER and KAHLMANN (*J. I. S. I.*, 1895, i., 512).—Absorb in Na_2O_2 in current of deoxidised air. Decompose Na_2O_2 by boiling and $KMnO_4$. Precipitate as usual.
891. ROCHOLL (*C. N.*, xlv., 236).—S may always be found in the residue, particularly when cupriferos irons are assayed.
892. BRUGMAN (*C. N.*, liv., 290).—Copper is no detriment to the evolution process, even when present up to 1 per cent.
893. BRAND (*J. I. S. I.*, 1884, 660).—Absorb in a burette of glass beads moistened with Br water; precipitate as usual.
894. TROILIUS (*J. I. S. I.*, 1884, 333).—Absorb in HCl+Br, pour into $BaCl_2$, and boil to drive off Br.
895. BLUM (*J. I. S. I.*, 1892, ii., 515).—When H_2S is absorbed as in 894, subsequent evaporation is necessary, not because Br interferes with the formation of $BaSO_4$, but in order to destroy oily hydrocarbons.
896. ROLLET (*J. I. S. I.*, 1880, 359) and GONTIERE (*J. S. C. I.*, 1896, 830).—The HNO_3 and $BaSO_4$ process is untrustworthy. Heat finely-divided metal, fuel, or slag in H (and CO_2). Collect H_2S in $AgNO_3$ and weigh as Ag_2S or Ag (see 935).
897. PHILLIPS (*J. S. C. I.*, 1896, 218).—The evolved H_2S is accompanied by organic sulphur compounds (methyl sulphide and mercaptan) not easily oxidised (see 908, &c.); pass through hot tube and absorb, in a capacious bottle, with HCl+Br.
898. TREADWELL (*C. N.*, lxiv., 217).—The insoluble sulphide is heated with Fe in CO_2 , the resulting ferrous sulphide treated with HCl, and the H_2S absorbed in $H_2O_2 + AmHO$. (*J. C. S.*, lxii., 1375).—Insoluble sulphides are quantitatively converted to H_2S by treatment with Sn and HCl without sulphates being attacked.
899. REED (*C. N.*, lxxii., 299).—Simple apparatus for absorbing H_2S in a small volume of liquid. Apparatus for same purpose by Blum (*J. C. S.*, lxii., 1376; *J. S. C. I.*, 1893, 1062), Meade (*J. S. C. I.*, 1897, 934), and Mackenzie (*J. S. C. I.*, 1893, 624).

II.b. Volumetric Estimation.—

900. ELLIOT (*C. N.*, xxiii., 61 and 143).—Absorb in NaHO, and determine H_2S in the acidified solution by titrating with iodine. The insoluble residue contains traces of S existing as sulphate.
901. PAYNE (*C. N.*, lxvi., 286) and LAUDIS (*C. N.*, lxxv., 218).—As 900. The standard iodine is prepared by adding a known amount of $KMnO_4$ to an acid solution of KI.
902. WILSON (*C. N.*, lxiv., 252).—The iodine is standardised by an actual sample containing a known amount of sulphur.
903. KOPFMAYER (*C. N.*, xxix., 134).—Pass evolved H_2S in a current of H into iodised KI, and titrate the excess of free I with hypo.
904. PETERS (*C. N.*, xxv., 11).—With white irons or steels all the S is not evolved as H_2S in 900, part remains with the residue (see 932).
905. HERTING (*C. N.*, lxxv., 109).—As Morrell (883), but titrate the CdS with iodine. It is unnecessary to expel air from the evolution flask with H or CO_2 .
906. TREADWELL (*J. I. S. I.*, 1893, i., 408) and CROBAUGH (*J. I. S. I.*, 1899, ii., 486).—Absorb in ammoniacal $CdCl_2$, acidify, and titrate with iodine.
907. THILL (*C. N.*, lxxii., 54).—Evolves H_2S into alkaline arsenite, pass acid vapours from evolution flask until As_2S_3 is precipitated, and titrate a filtered fraction of the excess As_2O_3 with iodine.
908. PROST (*J. I. S. I.*, 1889, i., 390).—Part of the S is present in cast-iron as an insoluble organic sulphur compound.
909. HERTING (*J. I. S. I.*, 1899, i., 484).—Resumé of gravimetric and volumetric processes. H_2S must

- be passed through a hot tube to decompose accompanying $(\text{CH}_3)_2\text{S}$ compounds.
910. CAMPREDON (C. N., lxxii., 299; and *J. I. S. I.*, 1897, ii., 503).—Evolve H_2S through heated tube along with H and CO_2 into ZnA_2 , and titrate ZnS with iodine. Time, fifteen to thirty minutes.
911. HOOFFER (C. N., lxxviii., 191).—Collect H_2S in NaHO, and titrate the alkaline solution with PbNO_3 .
912. HATTENSAUR (*J. S. C. I.*, 1891, 797).—Evolution with HCl gives for Siemens metal results identical with those gotten by 782.
913. BOUCHER (C. N., lxxv., 121).—Absorb in NaHO, add to Fe_2Cl_6 , and titrate FeO formed. Insoluble sulphides are treated with iron under charcoal, and estimated as above.
914. CAMMERER (*J. C. S.*, lxxi., 18 and 278) and HANNS (*J. C. S.*, lxxiv., ii., 461).—Most metallic sulphides react with Fe_2Cl_6 after the manner of 913.
915. WEIL (*J. C. S.*, l., 918).—Pass H_2S from mineral sulphide into AmHO solution of Cu and titrate the excess Cu in aliquot filtrate with SnCl_2 . The addition of Zn promotes the decomposition of the mineral.
916. FRIEDHEIM (*J. C. S.*, lii., 396, 618, and 998).—Process 915 is unreliable, because CuO goes down with the sulphide, and the latter tends to oxidise and re-dissolve. Weil subsequently absorbs in a mixture of CuSO_4 , NaK tartrate, and NaHO.
917. POPE (*J. C. S.*, lxxii., 123).—S is estimated in Ca_2C by decomposing with H_2O , then adding H_2SO_4 , and absorbing H_2S in PbA_2 .
918. KLOBULOW (C. N., liv., 325).—The presence of nascent H in the evolution flask converts SO_2 into H_2S , and may convert H_2SO_4 —or even S—thereto; therefore add Zn to the dissolving substance.
919. FRIEDMANN (*J. C. S.*, l., 739).—Process 918 is not generally applicable.
920. HERTING (*J. I. S. I.*, 1898, ii., 562).—To powdered blast-furnace slag add N/10 iodine, dilute HCl, and titrate unreacted I with hypo.
921. CRAIG (C. N., lxxiv., 266).—Sulphur exists in blast-furnace slags wholly as CaS , and may be determined as in 886, using purified coal-gas to sweep out, &c. Insoluble sulphides are heated with Zn dust. H_2S evolved and estimated as in 913.
922. EGGERTZ (C. N., xviii., 15).—Evolved H_2S colours Ag-Cu plate, which is then compared with standards. The amount of S in iron diminishes with time, at least on the surface.
923. RINMANN (*J. I. S. I.*, 1886, 397 and 1022).—Process 922 can be applied only to grey pig-iron; with white irons the results are very low. Möller says the test is quite untrustworthy.
924. OSMOND (C. N., lvii., 142).—Pass H_2S through a series of bulbs containing equal volumes of standard AgNO_3 . No H_2S passes any bulb until all the Ag is precipitated.
925. WIBORGH (C. N., liv., 158).—Pass H_2S through calico soaked in $\text{Cd}(\text{NO}_3)_2$, and compare colour with permanent standards made in the same way. Air must be driven from the flask prior to evolution of H_2S .
926. COHEN (*J. S. C. I.*, 1890, 16).—A simple form of apparatus for making Wiborgh's test.
927. MORGAN (C. N., lvi., 83).—Absorb H_2S in dilute PbA_2 , and compare colour with a series of standards.
928. ARNOLD and HARDY (C. N., lviii., 41, 59, and 71).—Process 927 gives PbS as a precipitate. Evolve H_2S in the presence of nascent H, collect in NaHO, add acidified PbA_2 , and compare tints. Or pass evolved H_2S through a series of PbA_2 tubes, as in 924, and count the number in which PbS is precipitated. Cu does not interfere.
929. LUCAS (*J. I. S. I.*, 1898, ii., 559).—Evolve H_2S by $\text{HCl} + \text{H}_2\text{SO}_4$ (H_2SO_4 alone gives low results), pass

- through hot tube into alkaline PbO, and estimate colorimetrically.
930. FUNK (*J. C. S.*, lxx., ii., 274).—Dissolve the zinc in HCl, absorb H_2S in AmHO + ZnSO_4 , add paramidodimethylaniline and Fe_2Cl_6 to the acidified solution, and compare the colour (blue) with standard solutions of H_2S . Can detect 0.0001 per cent sulphur.
931. SCHINDLER (*J. I. S. I.*, 1893, i., 408).—The HCl used should be concentrated in order to evolve all the S as H_2S .
932. WILLIAMS (*J. I. S. I.*, 1893, i., 409).—Less S is left in the residue when hot instead of cold HCl is used. One NaHO tube will absorb all the H_2S .
933. KONINCK (*J. I. S. I.*, 1895, ii., 597).—Add SnCl_2 or Sn to evolution flask along with the metal, to prevent oxidation of the FeO.
934. DONATH (*J. C. S.*, lxxiv., ii., 159).—An HCl solution of SnCl_2 reduces SO_2 to H_2S .
935. HOLLERT (C. N., xlvii., 13).—Heat to redness in a current of oxygen mixed with H and CO_2 ; collect evolved H_2S in AgNO_3 .
936. OTEHA (*J. I. S. I.*, 1898, i., 540).—Powdered coke mixed with Zn or Al and treated with HCl; S, evolved as H_2S , is estimated colorimetrically with CdA_2 .

III. MISCELLANEOUS ITEMS.

937. SCHLOSSBERGER (C. N., iii., 192 and 204).— Am_2MoO_4 in HCl gives a blue colour with H_2S or a metallic sulphide.
938. PRICE (C. N., viii., 285).—Any alkaline melt on the outside of a crucible readily absorbs S from a gas flame. The presence of Mn causes the fused salt to creep.
939. KONINGH (*J. C. S.*, lxxviii., ii., 184).—Sulphur may be oxidised to H_2SO_4 by boiling with KHO and permanganate.
940. MAR (C. N., lxxiii., 256).—Free HCl is favourable to the precipitation of Ba with H_2SO_4 , but it does not eliminate the contamination with alkaline salts, nor does dissolving in H_2SO_4 and diluting entirely, but evaporation of H_2SO_4 solution of BaSO_4 to crystallisation does.
941. BROWNING (C. N., lxxviii., 264).— HNO_3 and aqua regia are favourable to the formation of BaSO_4 , as in 940; the precipitate is more crystalline and is not prevented by citrate, &c.
942. JANNASCH (*J. C. S.*, lxxvi., ii., 60).—Se is separated from H_2SO_4 by boiling with hydroxylamine hydrochloride; Se is precipitated.
943. CARNOT and GOUTAL (*J. C. S.*, lxxii., ii., 555).—S exists in iron in combination with Mn by preference. On treating with Cu solutions, the sulphur combining with the Cu is equivalent to the Mn present in the steel.
944. SCHEUKER-KESTNER (*J. S. C. I.*, 1885, 281).—When Fe_2O_3 and CaSO_4 , MgSO_4 , or PbSO_4 are heated together, SO_3 is given off. Probably ferric sulphate is first formed and then decomposed at a higher temperature (see Jannasch, *J. S. C. I.*, 1889, 820); this does not occur with BaSO_4 .
945. ANON. (C. N., xix., 215).—By heating coke at 300° in a current of air at 2 to 3 atmospheres, all the sulphur is eliminated as SO_2 ; the calorific power of the coke is increased.

Analytical Research on some Essences of Jasmin. —MM. Jeancard and Satie.—To prevent the introduction of impurities into essence of jasmin, the authors use vaseline, unprepared by benjoin or orange-flower, instead of pork or beef fat. The perfume is then extracted in the known manner. The results are given of the analysis of several samples prepared in this manner.—*Bull. Soc. Chim.*, Series 3, vol. xxiii., No. 12.

ON THE TITRIMETRIC ESTIMATION OF THE MANGANESE IN MANGANATES BY MEANS OF ALKALINE SOLUTIONS OF ARSENIOUS ACID.

By T. REICHARD.

HAVING undertaken the examination of the manner in which different oxides behave in the presence of arsenious acid, I observed that manganic acid, when submitted to the action of an alkaline solution of arsenious acid, is immediately decomposed, even at the ordinary temperature. In the case of aqueous solutions, even when concentrated, it is necessary to use heat to start the reaction. This becomes manifest at about 50°.

By means of this reaction we are able to use arsenious acid for the estimation of the manganese in manganates, and for determining the oxidising power of these latter.

The decomposition takes place according to the following equation:—



This decomposition of the manganic acid has a double advantage from the analytical point of view. First of all, the arsenious acid, being easy to prepare in a state of perfect purity, can be utilised for the determination of the strength of the manganic acid. Then the manganous oxide set at liberty can be estimated gravimetrically, and thus constitutes a check on the results obtained by the titrimetric method. The indirect volumetric estimation of the manganese in manganates depends on the fact that a solution of arsenious acid of known strength can be compared with a solution of iodine. In a case when an excess of arsenious acid has been used, we can estimate the amount of unconsumed reagent by means of a solution of iodine. A simple calculation then enables us to find the amount of arsenious acid necessary to oxidise the manganic acid. As 5 molecules of arsenious acid are exactly equivalent to 4 atoms of metallic manganese, it is easy to find the amount of this latter body.—*Chemiker Zeitung*, xxiii., p. 801.

THE ESTIMATION OF GLYCERIN.

By CALIXTE FERRIER.

THE glycerin extracted from oils and fats is sold commercially under the name of glycerin from saponification. According to commercial custom it should have a density of 1.240, and should not leave more than 5 thousandths of ash or dehydrated incombustible material, after evaporation and calcination of the residue.

The verification of the density depends on methods which are too rigorous to leave any doubt about the results; but the same is not the case with regard to the estimation of the ash.

The results obtained in different commercial laboratories, from the same sample, are rarely concordant, and it is by no means uncommon to meet with differences of 20 or 30 per cent. It is thus evident that the method generally adopted is very defective.

As a rule we proceed in the following manner:—10 grms. of glycerin are evaporated in a platinum crucible, taking as much care as possible to prevent losses by projection. The tarry residue is then burnt in the air, and after extinction of the flame a further residue is left, consisting of carbon mixed with various salts. In order to consume the last traces of the carbon it is necessary to raise the temperature of the crucible to a very high point. The sulphates, chlorides, and alkaline carbonates which are generally found in the residue are melted, and are even volatilised, according to their respective volatilities, if the heating is too violent or too prolonged. It is even

possible for the whole of the alkaline chlorides to disappear before the carbon is entirely burnt away.

When we consider that the temperature is regulated only by the eye of the operator, it is easy to understand how such great divergences in the results are obtained.

It is certainly time to adopt a more trustworthy method, in order to do away with the numerous disagreements which occur. In our laboratory we make use of a method, which is a little longer it is true, but in which we have retained the great simplicity of the old method.

The glycerin is evaporated as has been described above. The tarry residue is burnt, and after roughly crushing the spongy mass which is left after combustion, we pour 5 or 6 c.c. of distilled water into the crucible, allow it to digest for a few moments, and then draw off the clear solution by means of a pipette with a capillary point, which does not allow the fragments of the carbonaceous residue to pass. Such a pipette is easily constructed by drawing out a glass tube in the blowpipe, and cutting off the end at the proper point to obtain a small opening. A second washing is then made in such a manner as not to use more than 10 or 12 c.c. of water altogether; this solution is retained.

The contents of the crucible are then dried and calcined at the temperature necessary for burning off the whole of the carbon. It must be observed that with this residue, from which all the soluble salts have been removed, the calcination is very rapid, and the disappearance of the carbon almost instantaneous.

After cooling, the wash-water is added to the calcined residue, and evaporated with the usual precautions. The crucible is then thoroughly dried, and raised to a red heat for a few seconds over a Bunsen flame.

By this method we have obtained constant results, exact to a ten-thousandth.—*Moniteur Scientifique*, December, 1900, p. 808.

ON THE OCCURRENCE OF TIN IN PRESERVED MEAT, TOGETHER WITH SOME OBSERVATIONS ON THE ESTIMATION OF, AND THE COMPOUNDS OF TIN IN PRESERVED MEAT.

By F. WIRTHLE.

SOME time ago I had occasion to search for tin in several samples of preserved meat, which had for a longer or shorter period (one to five years), been kept in tinned iron boxes. It was necessary at the same time to determine the state of combination of the tin absorbed by the meat, a problem which offered but little chance of success. An examination of the receptacles satisfied me that the layer of tin only contained a very small portion of lead (0.21 per cent); there was no soldered joint. In most cases the meat was separated from the juices containing the greater part of the fat, and these two portions were analysed separately. The results obtained are given in the accompanying table.

It will be seen that the amount of tin increases, as a rule, with the time of preservation, with the exception of the two-year old samples, and this might have been due to the nature of the meat. The quantity of tin is remarkably high in the case of the sample of meat preserved for five years, and the internal surface of the box was completely corroded. The meat absorbed three times as much tin as the juices, a fact similar to that recently observed by Günther (*Chemiker Zeitung*, 1900, xxiv., 50), in a sample of tinned herrings. To try and find the state of combination of the tin absorbed, the boxes were opened by a longitudinal incision, and their contents carefully removed.

I may here state that I observed that the interior of the

Contents of the box.	Length of time preserved. Years.	Number of samples examined	Tin, per cent.		Remarks.
			Meat.	Juices.	
Beef	1	2	Max. 0'0057 Min. 0'0039	0'0015 0'0014	
Goulasch	1	2	Max. 0'0051 Min. 0'0037	— —	
Beef	2	2	Max. 0'0036 Min. 0'0029	0'0016 0'0011	
Filet	2	1	0'0106	—	Box strongly attacked.
Beef	3	1	0'0074	0'0025	
Goulasch	3	1	0'0056	—	Box slightly attacked.
Filet	3	1	0'0079	0'0024	Do.
Beef	4	2	Max. 0'0082 Min. 0'0045	0'0028 0'0018	
Goulasch	4	2	Max. 0'0094 Min. 0'0061	— —	
Beef	5	4	Max. 0'0025 Min. 0'0088	0'014 0'0036	Box very strongly corroded.

boxes were corroded almost exclusively in the places in contact with the fat, while they were intact where there was an accumulation of gelatin. In the two boxes in which the meat had been kept for five years, we were able to detect a white crust on the corroded portions. This crust, though insoluble in water, dissolved easily on the addition of a drop of acid. The sulphuric or nitric solution of this white crust reduced corrosive sublimate and ammoniacal nitrate of silver, and gave a strong chlorine reaction; further, the tin was precipitated by sulphuretted hydrogen.

It is thus shown that the crust found in these tins is formed of basic chloride of tin, the formation of which was due to the action of the chloride of sodium present, on the tinned surface. It appears thus that chloride of sodium plays a prominent part in the corrosion of tinned iron boxes by preserved meats, either by direct action of the salt on the tin, or by first forming organic salts of tin, giving with the chloride of sodium basic stannous chloride and an organic salt of sodium. In the case of the four-year old preserved beef, the white crust was not observed, probably because the contents of the box were liquid. On the other hand, the whole of the interior surface of the box was covered with a thin black layer, which it was impossible to remove, giving sulphuretted hydrogen by the action of dilute acids, and probably formed of sulphide of tin. With the beef preserved for three years, the interior of the box was almost intact, and showed only traces of the white crust, which, dissolved in water containing a few drops of hydrochloric acid, and reduced solutions of mercuric chloride. I have not, however, been able to detect the presence of stannous compounds in boxes containing meat preserved for only one and two years.

With regard to the estimation of the tin in preserved meats and the destruction of the organic matter, I first made experiments with a view to oxidising the meat by means of chlorate of potassium and hydrochloric acid, but I did not obtain any good results. Although the residue from the first oxidation, consisting principally of fat, was oxidised a second time, the residuary fat still held notable quantities of tin. I did not think it advisable to use Kjeldahl's method for destroying the organic matter, as recommended by Halenke (*Chemiker Zeitung, Repert.*, 1899, xxiii., 99), because the amount of sample to be taken, viz., 120 grms., requires too large a quantity of sulphuric acid (400 c.c.). I finally adopted Orfila's process, which was recommended by K. B. Lehmann (*Archiv. Hygiene*, 1895, xxiv., 1), a process which I had already found to be of great service for the estimation of copper in preserved peas. The following is the method I finally adopted:—About 120 grms. of meat (the juices, separated from the meat and analysed apart, were treated in the same manner), were placed in a large porcelain dish of nearly 1 litre capacity, moistened with 5 c.c. of concentrated sulphuric acid, and heated carefully on a sheet of asbestos. It was frequently stirred, and from time to time small quantities

of sulphuric acid were added, altogether about 15 to 20 c.c.; the mass was occasionally removed from the sides of the crucible, to which it adhered, by means of a porcelain spatula. We thus obtained, after four or five hours, a porous carbonaceous mass, which was pulverised and incinerated in a porcelain crucible. The particles adhering to the porcelain dish were transferred to the crucible with the assistance of powdered anhydrous carbonate of soda; a further proportion of carbonate of soda was added, together with a sufficient quantity of nitrate of soda, the whole thoroughly mixed, and heated to gentle fusion. After cooling, the melted mass was taken up with water, and the cloudy solution obtained submitted to the action of a current of carbonic anhydride. When the cloudy solution had become quite clear (which generally takes place after about twelve hours), the precipitate was collected on a filter, well washed, dried, and incinerated. The ash was treated with a sufficient quantity of cyanide of potassium, and the mixture heated to dull redness, the crucible being closed. The melted mass was taken up with warm water, and the metallic tin and the iron collected on a filter, washed, and dissolved in a little warm hydrochloric acid. In the solution, which should not be very acid, the tin is precipitated with sulphuretted hydrogen, the precipitated sulphide is washed with water, saturated with sulphuretted hydrogen, and containing a small quantity of nitrate of ammonium, and then dried, incinerated, and calcined until the weight is constant.

The weighed stannic oxide is reduced once more by means of cyanide of potassium; the tin thus obtained is dissolved in hydrochloric acid, precipitated in the form of sulphide, and weighed as stannic oxide.—*Chemiker Zeitung*, 1900, p. 263.

THE ALLOTROPIC FORMS OF SELENIUM.*

By A. P. SAUNDERS.

(Continued from p. 298).

4. Red Crystalline Selenium.

THE methods by which this form may be obtained have been sufficiently illustrated in the description of the behaviour of the amorphous form in solvents. The direct change from the vitreous or amorphous modification to the red crystalline in the absence of solvents, has, as I have stated elsewhere, never been observed with certainty. There is no obvious reason why this change should not go on, and it is quite possible it could actually be observed by bringing the two forms in contact at a moderate heat.

As an exception to the general rule that selenium separates in the amorphous state when set free, it may be noted here that solutions of selenium in carbon disulphide throw down, on the addition of benzene, a shower of

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minute glittering plates, of pale red colour; the separation is not instantaneous.

The behaviour of vitreous selenium in carbon disulphide and Rammelsberg's determinations of the solubility of the amorphous form, point to the conclusion that the red crystalline form has, as we would expect, a lower solubility than those others. Exact measurements in this field are still wanting. The crystals which are obtained from carbon disulphide are very beautiful in form and colour, though small. They are readily broken under pressure, and yield a red powder which is indistinguishable from the amorphous modification. The red crystals, which have, in the mass, a dark maroon colour, are quite stable at ordinary temperatures. Mitscherlich, who first obtained and studied them, states that they are stable at 100° , but go over at 150° to the metallic form. Rammelsberg records later that they are instable even at 100° , but Muthmann (1891), who separated two distinct crystalline forms, places the limit of stability for the first, the ordinary form, at $110-120^{\circ}$, and for the second at $125-130^{\circ}$. The second form is, according to Muthmann, stable at 110° .

No further work has been done upon these two forms, the crystallographic properties of which will be considered below.

If their relationship to liquid selenium on the one hand, and to the metallic form on the other, is what I have supposed it to be, then the red crystals should have an instable melting-point below 217° . Mitscherlich records an observation bearing upon this, to the effect that these red crystals melt if rapidly heated to 200° .

This observation is perfectly easy to repeat, but it does not prove that the crystals have a melting-point at that temperature. We know, from Regnault's observations, that the heat of reaction in passing from the amorphous to the metallic form is large enough to raise the temperature of the selenium about 200° ; but since the red crystalline form is intermediate between these two, it is quite probable that a considerable quantity of heat is evolved when it is carried over to the metallic form; hence this apparently real melting-point may represent only what we may call a case of self-fusion, in which a part of the material in going over to the metallic form raises the temperature of the remainder to its melting-point. When some of the powder of the crystals is dropped on a platinum-plate, heated by means of an oil-bath to 170° or above, the powder melts, and this fusion is followed by an immediate transformation to the metallic form. Below 170° the change goes on more slowly and without fusion. By dropping the powder into a test-tube plunged in the bath, substantially the same results are obtained. Since the crystals are not themselves a form of the liquid modification, it is only necessary to show that, in melting, their temperature does not rise to 217° in order to establish the instable melting-point. For this purpose the powder of the crystals was mixed with some high-melting organic compounds, kindly supplied to me by Dr. C. E. Brewer, in this laboratory. The compounds used are:—Anisic acid, melting-point, 180° ; tri-methyl-acetyl-gallein, 196° ; and gallein acetate, 215° . The corresponding bath temperatures used were 170° , 190° , 205° respectively, and in no case was there evidence of fusion of the organic substance. The best results were obtained at 205° , the fusion of the selenium itself being less complete at the other temperatures; at this point the selenium was completely fused, whereas the crystals of the gallein acetate, although in intimate contact with the selenium, remained unchanged.

This experiment, taken in conjunction with the preceding one, shows that the red crystalline form has probably an instable melting-point at $170-180^{\circ}$.

The crystallography of this form of selenium was first studied by Mitscherlich (1855). He noted that selenium separates from carbon disulphide in two different crystal habits: (1) as thin, transparent, red, brilliant leaflets, and (2) as grains which are so intensely coloured as to appear opaque and almost black, although thin fragments of them

show the transparency and colour of the leaflets. He obtained the largest crystals by placing amorphous selenium together with carbon disulphide in a sealed tube, and subjecting this to successive changes of temperature between that of the room and a point somewhat below 100° . The crystals so obtained were scarcely 1 m.m. broad, but had such well-developed faces that it was possible to determine the angles by means of the goniometer.

Mitscherlich finds that the crystals are monoclinic; the better developed ones show a great variety of faces. He made a good many measurements of the angles, for which, however, the original article must be consulted.

Muthmann (1891), was able to separate two monoclinic forms, both obtained from carbon disulphide solutions; and he found that they possessed different degrees of stability. With regard to the form studied by Mitscherlich, Muthmann's results are confirmatory in the main. The axial relationships he found to be—

$$a : b : c = 1.63495 : 1 : 1.6095 \\ \beta = 75^{\circ} 58'.$$

The commonest faces are OP, ∞ P ∞ , +P - P, and ∞ P ∞ , though Muthmann also finds many other faces upon the better developed crystals. The colour is orange-red with a not very pronounced semi-metallic lustre.

The second form appears as short thick prisms with very pronounced semi-metallic lustre; they also are monoclinic, but the axial relationships are different—

$$a : b : c = 1.5916 : 1 : 1.352. \\ \beta = 86^{\circ} 56'.$$

The observed faces were ∞ P, OP, ∞ P ∞ , and P ∞ . This form comes out sometimes as leaflets, which are then easily confused with crystals of the first form. The colour is deep red.

(To be continued).

INAUGURATION OF MEMORIAL BUST OF DR. E. DIVERS.

A LARGE and interesting meeting was held on Saturday, November 17, in the ground of the Tokyo Imperial University, to inaugurate the memorial bust of Dr. Edward Divers, erected by his friends and former pupils, in front of the Science College, in grateful remembrance of his long and important services in Japan. Past and present Presidents of the University, most of the Professors, and a large number of Dr. Divers' former pupils attended the meeting, whilst Mr. E. W. Tilden, son-in-law of Dr. Divers, came up from Kobe specially for the occasion.

The ceremony was opened at 11.30 a.m. by an address by Prof. J. SAKURAI, who represented the Committee, and who dwelt, at some length, upon the important services done by Dr. Divers, both in the former Engineering College and the present Science College, during the past quarter of a century, and both in the capacity of a teacher and that of an investigator. The bust was then unveiled amidst loud cheers, and presented to the University.

Dr. D. KIKUCHI, President of the University, next read an address, in which he referred to the high recognition by the University of the important works done by Dr. Divers for the cause of education and science, and expressed the pleasure, in his official capacity, of accepting the bust.

Mr. E. W. TILDEN, on behalf of Dr. Divers and his family, briefly thanked for the courteous way in which the Doctor's friends and former pupils expressed their appreciation of his services.

The ceremony was concluded by a speech by Mr. H. WATANABE, one of the oldest friends of Dr. Divers, and who referred to his official connections with the learned Doctor both at the old Kobudaigakko and the Imperial University, and expressed his admiration of the long and unwearied work of Dr. Divers.

Music was played by the Tokyo City Band between the addresses and speeches, and afterwards refreshments were served at Dr. Divers' former residence.

The bust, which was executed by Mr. Naganuma, won an unanimous approbation.

THE REDUCTION OF SELENIUM DIOXIDE BY SODIUM THIOSULPHATE.

By JAMES F. NORRIS and HENRY FAY.

SOME time ago we proposed a volumetric method for the estimation of selenous acid (*Amer. Chem. Journ.*, xviii., 703) based on the reaction between it and sodium thiosulphate in the presence of hydrochloric acid. The procedure, in brief, is as follows:—The solution in which the selenous acid is to be determined is diluted with ice-water, acidified with dilute hydrochloric acid, and an excess of a tenth-normal solution of sodium thiosulphate added. The excess of thiosulphate is determined by titration with a solution of iodine. It was shown that 1 molecule of selenous acid reacted with 4 molecules of sodium thiosulphate, and that the reaction afforded a means for the accurate determination of selenium.

At the time this method was proposed we were not able to state the exact nature of the reaction, but we have studied further the reduction, and are now able to write the complete reaction. The most probable reaction between the two substances may be represented by the following equation:—

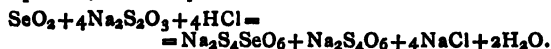


If strong solutions of selenium dioxide and sodium thiosulphate are mixed, selenium is precipitated, and the solution shows an alkaline reaction. In dilute solutions, on the other hand, no selenium is precipitated, and the reaction is not complete according to the above equation, since the sodium hydroxide formed neutralises a part of the selenous acid which, accordingly, does not enter into the reaction. If, however, acid is present, 4 molecules of sodium thiosulphate react with 1 molecule of selenium dioxide. The amount of acid required to complete the reaction was determined. This was found to vary with the dilution, the excess of thiosulphate, and the time of reaction. The following table gives the results obtained. In each case 10 c.c. of a solution of selenium dioxide, which was equivalent to 26.40 c.c. of sodium thiosulphate, according to the ratio $\text{SeO}_2 : 4\text{Na}_2\text{S}_2\text{O}_3$, were used.

	Molecules HCl.	$\text{Na}_2\text{S}_2\text{O}_3$ taken. C.c.	$\text{Na}_2\text{S}_4\text{O}_6$ used. C.c.	Water added. C.c.
I.	3	50	17.90	—
II.	4	27	22.02	300
III.	4	27	24.96	300
IV.	4	50	25.40	300
V.	5	30	26.38	300
VI.	4	50	26.40	—

In Experiment I., 3 molecules of acid were used, and only 17.90 c.c. of thiosulphate entered into the reaction. Using 4 molecules in II., 22.02 c.c. of thiosulphate reacted. In the third experiment the same proportions were used, but the solutions stood forty minutes before the excess was determined. This had a marked effect on the result, as about 3 c.c. more of the thiosulphate reacted. A large excess of thiosulphate hastened the reaction, as is shown in Experiment IV. Experiment V. shows that 5 molecules of acid are sufficient at a dilution of 300 c.c., and with a slight excess of sodium thiosulphate, to complete the reaction. In Experiment VI. no water was added to the solutions used, and the reaction was complete when the proportion of the reagents were $\text{SeO}_2 : 4\text{Na}_2\text{S}_2\text{O}_3 : 4\text{HCl}$. These are, therefore, the amounts of reagents which enter into the reaction.

In order to determine the products of the reaction, a solution of the constituents in the above proportions was prepared. The study of this solution led to the following equation, which expresses the reaction:—



The formation of sodium selenopentathionate, similar to potassium pentathionate, seemed probable, since no selenium was precipitated as a result of the reduction. Debus (*Journ. Chem. Soc.*, liii., 278), in an exhaustive study of Wackenroder's solution, which is prepared by the action of hydrogen sulphide on an aqueous solution of sulphur dioxide, isolated some salts of pentathionic acid in a pure condition, and carefully studied their properties. He gives the following characteristic reactions:—

"I. An ammoniacal solution of silver nitrate causes, in a solution of potassic, ammonic, or baric pentathionates, a brown colouration which rapidly becomes darker, and by degrees a black precipitate is thrown down from the mixture. This reaction is not produced in a solution of tri- or tetrathionates, potassic thiosulphate, or ammonic sulphite. An ammoniacal solution of silver nitrate also seems to have no effect on them. Consequently a pentathionate, even if present in very small quantity, can be detected by means of this reaction in a mixture containing potassic tri- and tetrathionates, and sodic, potassic, or ammonic thiosulphates.

"II. Potassic hydroxide, in solutions of pentathionates, immediately produces a separation of sulphur. As tri- and tetrathionates and thiosulphates are not changed by this reagent, a proportionally small quantity of a pentathionate can be detected in a mixture of the four salts by addition of potassic hydroxide.

"III. Ammonia added to a solution of potassic pentathionate causes, after about one or two minutes, a precipitation of sulphur.

"IV. Hydric chloride does not change solutions of tetra- and pentathionates."

A solution prepared by mixing the constituents in the proportions represented in the equation was subjected to the above tests. A precipitate was formed with ammoniacal silver nitrate, as described above under I., and potassium hydroxide caused an immediate precipitation of selenium. Ammonia acted more slowly, and the solution was stable toward strong hydrochloric acid. The solution showed, therefore, the characteristic reactions of a penthionate, selenium, however, being precipitated instead of sulphur. This is in accord with the formula proposed, $\text{Na}_2\text{S}_4\text{SeO}_6$. An effort was made to isolate the selenopentathionate, but without success, as selenium was always precipitated when the solutions were concentrated by heat or in a vacuum. From the solution, however, we were able to isolate sodium tetrathionate, which was proved to be such by qualitative and quantitative analysis.

A dilute solution containing the new salt could be boiled some time without change. A number of neutral salts were found to decompose the compound. Stannous chloride caused a precipitation of selenium after a few minutes. A dilute solution of sodium thiosulphate produced the same effect, only much more slowly, whereas a number of hours' standing was necessary before potassium iodide caused any decomposition.

An acid solution of tellurium dioxide is reduced by sodium thiosulphate, giving a bright yellow solution, from which sodium hydroxide precipitates tellurium. Tellurium, therefore, probably forms a compound analogous to a selenopentathionate. This is remarkable, since no compounds containing tellurium, analogous to the thionates, are known. Crane (Johns Hopkins University) attempted to prepare substances similar to the thiosulphates, by boiling sodium tellurite with sulphur, selenium, and tellurium, but there was apparently no reaction.

By misunderstanding a statement made in our former paper, Mr. J. T. Norton, Jun. (*Amer. Journ. Sci.*, clxvii., 287), has been led to study the influence of hydrochloric

acid on solutions with sodium thiosulphate, and to repeat our work on the estimation of selenium. He says, referring to the method, "the explicit statement of the authors that the amount of hydrochloric acid present does not influence the result provided the titration is made at the temperature of melting ice, is an extraordinary view in view of generally accepted ideas as to the interaction of hydrochloric acid and sodium thiosulphate, as to suggest the necessity of a careful investigation of this point." In the paper referred to we made this statement:—"It was found necessary to have enough hydrochloric acid present to set free all of the thiosulphuric acid. If the solution is cold a large excess of hydrochloric acid does not affect the titration." Mr. Norton evidently confuses excess with amount. We used in all of our experiments 10 c.c. hydrochloric acid (1.12 sp. gr.), which is seven to eight times the amount required according to the above statement, and obtained excellent results. The statement is, therefore, not so extraordinary as might appear.

As the action of hydrochloric acid on sodium thiosulphate was known to us when we were making a study of the method, we took the precautions to have the solutions containing the acid dilute, and used ice to keep the temperature as low as possible. The necessity for these precautions was stated in the directions given. Mr. Norton further points out that it is advisable to use not more than 20 c.c. excess of sodium thiosulphate in order to prevent decomposition by the acid. We have made some new determinations of selenium in order to test the directions given in the original paper under the most unfavourable conditions. The results follow:—

	SeO ₂ taken. Gm.	Na ₂ S ₂ O ₃ taken. C.c.	Na ₂ S ₂ O ₃ used in reaction. C.c.	SeO ₂ found. Gm.	Dilution. C.c.
I.	0.0095	43	39.62	0.0095	300
II.	0.0336	15	13.39	0.0336	300
III.	0.0336	50	13.13	0.0331	300
IV.	0.0336	50	13.24	0.0333	300
V.	0.0435	50	17.20	0.0432	400
VI.	0.0430	50	16.41	0.0412	200

In Experiments I. and II. the directions as given in our paper were followed, and a small excess of sodium thiosulphate was used. In order to have a large excess of thiosulphate present in the other experiments, small amounts of selenium dioxide and 50 c.c. of thiosulphate were used. In III. and IV. the excess, 36.87 c.c., was nearly three times the amount necessary for the reaction—13.13 c.c. Since a bare excess only is necessary, a careful analysis would hardly be based on such a procedure. Experiment VI. shows the necessity of working in dilute solutions. In all of the above determinations 10 c.c. hydrochloric acid (1.12 sp. gr.) were used, but since 5 c.c. is quite sufficient to bring about the reaction, the modification which Mr. Norton suggests as the result of his work, namely, the use of the latter amount of acid, can readily be accepted.

Mr. Norton points out also that his results always come high. We had not observed this in our work, and accordingly we sought the cause. As we had standardised the sodium thiosulphate by titration against known weights of iodine, under the conditions which were to be used in the subsequent analyses—that is, titration in the cold in the presence of acid—a standardisation was made under ordinary conditions. The factor found was higher by about 0.2 per cent than the one previously obtained. This discrepancy was shown to be due to the fact that the iodine-starch reaction is more sensitive at 3° in presence of dilute acids than under the conditions which are ordinarily used in the titration of iodine and thiosulphate. The following table gives the number of cubic centimetres of one-hundredth normal iodine solution required to produce a colour with starch under different conditions of temperature and with varying amounts of dilute hydrochloric acid (sp. gr. 1.12). The starch solution was made by grinding 2 grms. of soluble starch with 5 c.c. of cold

water, and pouring the mixture into 500 c.c. boiling water. In all tests 5 c.c. of this solution were used and diluted to 300 c.c. with water.

No.	Acid C.c.	Temperature.	Iodine.
1	—	20°	0.61
2	1000	20°	0.32
3	1000	20°	0.26
4	5000	20°	0.27
5	—	3°	0.30
6	—	15°	0.30
7	—	20°	0.37
8	—	25°	0.35
9	—	30°	0.17
10	1000	3°	0.15

Experiments 1 to 4 show the effect of acids and 5 to 9 the effect of temperature. In Experiment 10 the most favourable conditions for the reaction were combined and 0.15 c.c. of the iodine solution gave a distinct colour, whereas in the absence of acid, at 25°, 0.35 c.c. was necessary. It has long been known that temperature has an effect on the line compound formed by the action of iodine on starch, but, as far as we can find, it has never been shown that this effect is appreciable at the temperatures used in the course of an analysis. Although the error arising is small when the facts brought out by the above experiments are overlooked, nevertheless, when very accurate results are desired, they should be taken into consideration.—*American Chemical Journal*, xxiii., No. 2.

PROCEEDINGS OF SOCIETIES

CHEMICAL SOCIETY.

Ordinary Meeting, December 6th, 1900.

Professor THORPE, C.B., F.R.S., President, in the Chair.

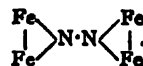
(Concluded from page 300.)

162. "The Heat of Formation and Constitution of Iron Nitride." By GILBERT JOHN FOWLER, M.Sc., and PHILIP J. HARTOG, B.Sc.

To determine the heat of formation of iron nitride, the substance was dissolved in dilute sulphuric acid (containing 49 grms. to the litre), as represented by the equation—



A current of nitrogen was passed through the solution to avoid oxidation of the ferrous salt, as the reaction is a slow one, though it eventually becomes complete. The mean of three determinations gave as the thermal value of the reaction +81.56 cal. From this value the heat of formation of iron nitride is calculated to be +30.4 cal. It is suggested that the constitution of iron nitride is—



163. "Infracampholenic Acid: an Isomeride of Campholytic and Isolauronic Acids." By M. O. FORSTER.

By hydrolysing the unsaturated nitrile formed when the anhydride of bromonitrocamphane is treated with hot aqueous sodium hydroxide (*Trans.*, 1899, lxxv., 1141), *infracampholenic acid*, C₉H₁₄O₂, is obtained as a viscous oil boiling at 145° and 239°, under pressures of 24 m.m. and 758 m.m. respectively; it is optically inactive, and is transformed into isolauronic acid by hot, dilute, sulphuric acid. The *dibromide*, C₉H₁₄O₂Br₂, produced when a cold solution of bromine (1 mol.) is added to the acid dissolved in chloroform and surrounded by a freezing mixture, crystallises from warm petroleum in minute white needles, which melt at 125° to a colourless liquid

which evolves gas. *Tribromodihydroinfracampholenic acid*, $C_9H_{13}O_2Br_3$, obtained when infracampholenic acid is submitted to the action of bromine under the conditions which lead to the conversion of campholytic acid into its dibromide, crystallises from ethyl acetate, and melts at 182° to a colourless liquid which evolves gas.

Aminoinfracampholenic acid, $C_8H_{13}NH_2$, prepared from the amide of infracampholenic acid by the action of sodium hypobromite, is a colourless oil which boils at $158-160^\circ$ under 754 m.m. pressure, and has a sp. gr. 0.8770 at 14° . The *hydrochloride* melts at 213° , the *platinichloride* at $238-240^\circ$, and the *picrate* at 213° ; the *benzoyl carbamide*, and *phenylcarbamide* derivatives melt at 105° , 182° , and 180° respectively.

164. "Tutu." Part I. By THOMAS HILL EASTERFIELD and BERNARD CRACROFT ASTON.

The authors have examined three varieties of tutu—*Coriaria ruscifolia*, *C. thymifolia*, *C. angustissima*—which cause serious loss of stock in New Zealand, and have isolated from them a glucoside, *tutin*, $C_{17}H_{20}O_7$, along with acetic, gallic, succinic, and other acids. From *C. thymifolia* quercetin or an isomeric compound was obtained; and from *C. angustissima* a volatile acid, $C_8H_8O_4$, which has not been identified.

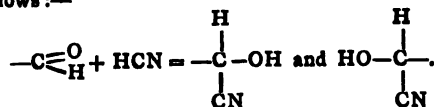
Tutin was obtained in colourless crystals melting at $208-209^\circ$ (uncorr.), and is perceptibly volatile at $120-130^\circ$. Its solubility is 1.9 grms. in 100 grms. water at 10° ; 1.5 grms. in 100 grms. ether at 10° ; 8.2 grms. in 100 grms. alcohol at 16° . It is very soluble in acetone, sparingly soluble in chloroform, insoluble in benzene and carbon disulphide. Its optical rotation was determined, and gives $[\alpha]_D^{19} = +9.25^\circ$.

The authors also discussed the formula for coriamyrtin, and conclude that the formula, $C_{15}H_{18}O_5$, which is half that usually accepted, harmonises better with the facts. A comparison of the chemical and physical characters of tutin and coriamyrtin proved that these substances are not identical.

A note by Professor Marshall on the pharmacology of tutin states that its action is similar to that of coriamyrtin, but is more slowly produced, and it is much less toxic. Its action is exerted mainly on the medulla oblongata and the basal ganglia of the brain.

165. "Experiments on the Production of Optically-active Compounds from Inactive Substances." Preliminary Notice. By J. B. COHEN and C. E. WHITELEY.

E. Fischer has shown (*Annalen*, 1892, cclxx., 64) that in the synthesis of one sugar from another by the addition of hydrocyanic acid to the lower member, a new asymmetric carbon atom is introduced, which may give rise to two stereoisomeric compounds. This may be represented as follows:—



Although the two new groups are optical antipodes, the two isomerides are not necessarily so as a whole, and consequently they are not always produced in equal quantities. Fischer has found, for example, that glucose forms two cyanhydrins in very unequal quantities, whilst in the case of dextro-mannose only one of the two possible cyanhydrins is produced (Hartmann, *Annalen*, 1892, cclxxii., 190). It is clear, therefore, that the asymmetric molecule as a whole exerts its influence on the space configuration of the newly-added asymmetric carbon group. In these examples, it is impossible to determine exactly the influence of the active part of the original molecule on the activity of the new group, seeing that the latter cannot be detached from the molecule. As the formation of active substances in living organisms is probably closely connected with their production from other active substances, from which they are afterwards removed, the idea occurred to us to attempt to produce a new asym-

metric carbon atom in an already active compound from which the originally active group could be subsequently detached.

A number of reactions readily presented themselves, such as the reduction, bromination, or hydroxylation of esters composed of an unsaturated acid and an active alcohol, or the reduction of a ketonic ester of an active alcohol, the alcohol being afterwards removed by hydrolysis.

These reactions may be represented as follows: (X stands for an atom or group not being hydrogen, A indicates the active alkyl or aryl group, and C the new asymmetric carbon atom):—

1. $\begin{cases} (a) \cdot CH:CH \cdot CO_2A. \\ (b) \cdot CH_2 \cdot CHX \cdot CO_2A. \\ (c) \cdot CH_2 \cdot CHX \cdot CO_2H. \end{cases}$
2. $\begin{cases} (a) \cdot CH:CH \cdot CO_2A. \\ (b) \cdot CHX \cdot CHX \cdot CO_2A. \\ (c) \cdot CHX \cdot CHX \cdot CO_2H. \end{cases}$
3. $\begin{cases} (a) \cdot CO \cdot CO_2A. \\ (b) \cdot CHOH \cdot CO_2A. \\ (c) \cdot CHOH \cdot CO_2H. \end{cases}$

Under (1) we have studied the reduction of the menthyl esters of mesaconic and phenylcrotonic acids.

- (a) $CH_3 \cdot C(CO_2C_{10}H_{19}):CH \cdot CO_2C_{10}H_{19}.$
- (b) $CH_3 \cdot CH(CO_2C_{10}H_{19}):CH_2 \cdot CO_2C_{10}H_{19}.$
- (c) $CH_3 \cdot CH(CO_2H)CH_2 \cdot CO_2H.$
- (a) $C_6H_5 \cdot CH:CH(CO_2C_{10}H_{19}) \cdot CO_2C_{10}H_{19}.$
- (b) $C_6H_5CH_2 \cdot CH(CO_2C_{10}H_{19}) \cdot CO_2C_{10}H_{19}.$
- (c) $C_6H_5CH_2 \cdot CH(CO_2H) \cdot CO_2H.$

Under (2) we have prepared the bromine derivatives of the amyl and menthyl esters of cinnamic acid and of dicinnamyl tartrate, and examined the action of various reagents on the dibromo-compounds, also the action of sodium ethylate on menthyl fumarate.

- (a) $C_6H_5CH:CH \cdot CO_2C_5H_{11}.$
- (b) $C_6H_5CHBr \cdot CHBr \cdot CO_2C_5H_{11}.$
- (a) $C_6H_5CH:CH \cdot CO_2C_{10}H_{19}.$
- (b) $C_6H_5CHBr \cdot CHBr \cdot CO_2C_{10}H_{19}.$
- (a) $C_6H_5CH:CH \cdot CO_2 \cdot CH \cdot CO \cdot O.$
- (b) $C_6H_5CHBr \cdot CHBr \cdot CH \cdot CO_2H.$
- (a) $C_6H_5CHBr \cdot CHBr \cdot CH \cdot CO_2H.$
- (b) $CH \cdot CO_2 \cdot C_{10}H_{19}.$
- (a) $||$
- (b) $CH \cdot CO_2 \cdot C_{10}H_{19}.$

Under (3) we have investigated the reduction of menthyl pyruvate.

- (a) $Me \cdot CO \cdot CO_2C_{10}H_{19}.$
- (b) $Me \cdot CHOH \cdot CO_2C_{10}H_{19}.$
- (c) $Me \cdot CHOH \cdot CO_2H.$

The results in all cases have been of a negative character, in spite of every care to avoid possible racemisation by conducting the reactions as far as possible at the ordinary temperature.

The experiments, which were commenced in 1898, have been delayed in consequence of unforeseen difficulties. All the experiments under (2), after a considerable loss of time, had to be abandoned. Although the dibromo-derivatives of amyl and menthyl cinnamate and of dicinnamyl tartrate could be readily obtained in a state of purity, they could not be directly hydrolysed without removing bromine, and all attempts to replace the bromine by hydroxyl failed. Menthyl fumarate, which was readily prepared from silver fumarate and menthyl chloride, gave no ethoxy-derivative by means of Purdie's reaction. Reactions (1) and (3) were, however, ultimately brought to a satisfactory conclusion.

Menthyl mesconate and menthyl phenylcrotonate were

prepared by heating together menthol and the acid chloride, whilst menthyl pyruvate was obtained by heating menthol and pyruvic acid under diminished pressure. The reduction of the first two esters was effected by means of the aluminium-mercury couple in aqueous alcoholic solution, whilst menthyl pyruvate was reduced with zinc dust and acetic acid. The esters were then hydrolysed with alcoholic potash and the menthol removed with ether. The acid, after careful purification by crystallisation or precipitation of its salt, was finally examined in the polarimeter. Pyrotartaric acid, phenylmethylacetic acid, and lactic acid, obtained in this way, were all optically inactive. We propose to extend these experiments to the acid amides of optically active bases.

166. "Synthesis of Isocamphoronic Acid." By W. H. PERKIN, Jun.

When the anhydride of $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, *aa*-dimethylglutaric acid, is treated with phosphorus pentachloride and bromine, and the product poured into alcohol, $\text{CO}_2\text{Et}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{CHBr}\cdot\text{CO}_2\text{Et}$, *ethylbromo-dimethylglutarate* is obtained as a colourless oil boiling at $165-170^\circ$ (35 m.m.).

This, on treatment with alcoholic potash, yields *dimethylglutaconic acid*, $\text{CO}_2\text{H}\cdot\text{CMe}_2\cdot\text{CH}:\text{CH}\cdot\text{CO}_2\text{H}$, which crystallises from water in plates melting at 172° , and is possibly a stereoisomeride of the *aa*-dimethylglutaconic acid lately prepared by M. Conrad (*Ber.*, 1900, xxxiii., 1920), which melts at 150° . That the acid of melting-point 172° has the constitution assigned to it is proved by the fact that when oxidised with permanganate at 0° it is quantitatively converted into dimethylmalonic acid and oxalic acid,—



Dimethylglutaconic acid is readily esterified by means of alcohol and sulphuric acid: the *ethyl*dimethylglutaconate, $\text{CO}_2\text{Et}\cdot\text{CMe}_2\cdot\text{CH}:\text{CH}\cdot\text{CO}_2\text{Et}$, thus produced, is a colourless oil boiling at 200° (200 m.m.).

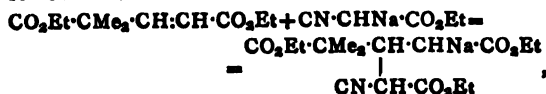
When this ester is digested in alcoholic solution with the sodium compound of cyanacetic ester, condensation takes place, and the product, on heating with dilute sulphuric acid, is almost quantitatively converted into an acid melting at 166° , and which on examination has been proved to be *isocamphoronic acid*.

(Found: C=49.7; H=6.5; $\text{C}_9\text{H}_{14}\text{O}_6$ requires C=49.5; H=6.4).

Professor von Baeyer was kind enough to send the author a sample of isocamphoronic acid which had been prepared from pinene, and on mixing equal quantities of this and the synthetical acid no alteration in the melting-point could be observed. Again, when heated with sulphuric acid at 100° , the synthetical acid is converted into *terpenylic acid*, a change which Tiemann (*Ber.*, 1896, xxix., 2613) has shown to be characteristic of isocamphoronic acid.

There can thus be no doubt that the synthetical acid is in reality isocamphoronic acid, and its synthesis solves the vexed question of the constitution of this important acid.

The condensation of ethyl dimethylglutaconate with the sodium compound of ethyl cyanacetate may be represented thus:—



and the cyanester thus produced yields, on hydrolysis and elimination of carbon dioxide, isocamphoronic acid, which, therefore, must have the constitution,—



first proposed for this acid by Tiemann (*Ber.*, 1896, xxix., 2614).

167. "On some α -Alkyl Substitution Products of Glutaric, Adipic, and Pimelic Acids." By J. W. MELLOR.

The author describes the known methods of general application for the preparation of the above acids, and gives an account of some new methods which he has employed in synthesising such acids. He finds that adipic acid itself may be readily prepared in quantity by the action of *chloropropylcyanide*, $\text{Cl}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CN}$, on the sodium compound of ethyl malonate, and subsequent hydrolysis of the *ethylcyanpropylmalonate*,—



(b.p. $170-175^\circ$ at 40 m.m.), by boiling with dilute sulphuric acid. α -Alkyl substitution products of adipic acid may be obtained by employing the corresponding ethyl α -alkyl malonates in this synthesis in the place of ethyl malonate.

The author has prepared several of the known α -alkyl substitution products of glutaric, adipic, and pimelic acids in a state of purity, and determined their dissociation constants. He has also prepared α -propyl adipic acid, $\text{CO}_2\text{H}\cdot\text{CHPr}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{H}$, which melts at 59° , and does not appear to have been previously described.

168. "On the Nature of Polyiodides and their Dissociation in Aqueous Solutions." By H. M. DAWSON.

From experiments on the ratio of distribution of iodine between solutions of potassium iodide and carbon bisulphide, and the simplicity of the dissociation isotherm for the equilibrium in the aqueous solution, which accords with the experimental data, it is concluded that potassium triiodide is a normal salt of potassium, which at corresponding concentrations is dissociated electrolytically to the same extent as other binary salts. Experiments with solutions of hydriodic acid lead to the conclusion that hydrogen triiodide is electrolytically dissociated to the same extent as hydriodic acid, and belongs, therefore, to the group of strong acids. These results are in harmony with Abegg and Bodländer's theory of complex compounds. From their analogy with potassium triiodide, the recently discovered trihaloid derivatives of caesium and rubidium must be placed in the same category

NOTICES OF BOOKS.

A Manual of Assaying: the First Assay of Gold, Silver, and Lead, including Amalgamation and Chlorination Tests. By ALFRED STANLEY MILLER. New York: John Wiley and Sons. London: Chapman and Hall, Limited. 1900. 87 pp. 12 mo. Illustrated.

In the Preface to this little volume the author explains that it departs from the plans of other books, by introducing the apparatus where its use is easily understood. "The student is taught the operations of the fire-assay by practising with an ore easy to assay; he is then better prepared to understand the directions for assaying more complex ores." In the first chapter the student is told how to make cupels and flux; in the second, how to prepare a sample of ore, and how to make an assay of a siliceous gold and silver ore.

The instructions are clear, and the many illustrations aid in their comprehension. The author cites standard works on the subject. There is an index.

H.C.B.

Chemistry for Examinations. By HENRY W. HILL. London: Allman and Son. 1900. Pp. 130.

As examinations are a necessary evil, it follows that books will be published with the sole object of helping the idle student to "cram"; a diligent man has no need for such artificial help.

The book now before us has been written more particularly to meet the requirements of the Oxford and Cambridge Local Examinations; and we further notice that, so as to prevent younger men overloading their minds, all sections

which are of importance to senior students only have been marked with an asterisk.

Considering its avowed object, this volume is well planned, and covers a fairly wide range of elementary theoretical chemistry. It also contains a "Special Part" which includes sections devoted to chemical diagrams and equations, and to several original tables, notably comprehensive tables of colour and solubility.

CORRESPONDENCE.

DETECTION OF ARSENIC.

To the Editor of the Chemical News.

SIR,—The difficulty that Mr. Estcourt experienced when trying to detect arsenic in Manchester beer without previous treatment by means of Marsh's test, is one that is likely to occur when any substance containing sulphites and sulphides, or any other compound that contains sulphur that is converted into sulphuretted hydrogen by zinc and acid, is examined by means of Marsh's test for arsenic.

The reason of this was pointed out indirectly by Otto Fehr and der Plordten in a paper on the "Removal of Arsine from Sulphuretted Hydrogen," published in the *Ber.*, xvii., 2897, and abstracted in the *Journal of the Society of Chemical Industry* for 1885, and no doubt in other journals and papers. The reactions bearing on the above subject mentioned by him are as follows:—

The decomposition of sulphuretted hydrogen at temperatures from 350° to 400° C. The decomposition of arseniuretted hydrogen at higher temperatures, and the mutual reaction of arsine in sulphuretted hydrogen between 350° and 360° C., which reaction he says is not complete at this temperature.

I have no doubt that if Mr. Estcourt analyses the substance he obtained in heated tubes he will find that it was a mixture of free sulphur and sulphide of arsenic, which I have frequently got when testing similar mixtures.—I am, &c.,

JAS. F. SMITH.

30, Chester Road, Akroydon, Halifax.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxxi., No. 23, December 3, 1900.

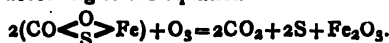
Samarium Carbide.—Henri Moissan. —Samarium oxide, at the temperature of the electric furnace and in presence of carbon, forms a crystallised carbide of formula SaC_2 . The composition of this carbide is comparable with that of the carbides of cerium, lanthanum, neodymium, and praseodymium. It decomposes cold water in the same way as the carbides of the alkaline earths, giving a complex mixture of hydrocarbons very rich in acetylene. It has a density of 5.86, a yellow colour, and, when examined under the microscope, has a crystalline appearance—the particles having an hexagonal shape. This substance burns brilliantly at 400° in a current of oxygen. The decomposition of water by the carbide brings the metal samarium nearer to the yttrium group, and removes it further from the rare earths belonging to the cerium group.

Acidimetry of Aldehydes and Acetones.—A. Astruc and H. Murco.—M. Astruc has already examined the

alkalimetry and acidimetry of the amines, phenols, and organic acids. The authors now examine the reactions furnished by aldehydes and acetones in presence of the colour reagents—helianthine, phenolphthalein, and Poirrier's blue. The following substances were examined:—Monoaldehydes of simple function, dialdehydes of simple function, halogen aldehydes, alcohol and phenol aldehydes, monoacetones of simple function, diacetones, halogen acetones, alcohol acetones, and acid acetones. The authors propose to confirm and extend their researches relative to the aldehydes and acetones by the thermochemical examination of these bodies, which is as yet very incomplete.

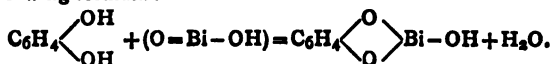
Some Reactions of Substituted Anilines.—E. Chasner de Coninck.—The author's experiments were performed on extremely pure specimens of methyl, dimethyl, ethyl, and diethyl anilines. The reactions of these substances were noticed with cupric chloride solution, copper sulphate solution, and copper acetate solution, dilute solution of cobalt chloride, dilute solution of nickel chloride, and both these solutions when concentrated. A careful investigation of all these reactions gives a means of identifying and distinguishing the various anilines, but the reactions are apparently masked by impurities.

Presence of Oxysulphocarbonate of Iron in the Water of the Rhone.—H. Causse.—Rhone water, during a period of three months commencing in June, contains iron oxysulphocarbonate. The proportion of this compound, very slight at first, increases progressively until the middle of September, then decreases and disappears in the autumn. The origin of this oxysulphocarbonate of iron may possibly be due to the combination of carbon dioxide with the ferrous sulphide due to the reduction of sulphates by certain organic matters in the river, according to the equation $\text{CO}_2 + \text{FeS} = \text{CO} < \text{S} > \text{Fe}$. In contact with air, this compound appears to be destroyed by the oxygen, according to the equation—



Journal de Pharmacie et de Chimie,
Series 6, Vol. xii., No. 4.

Some Bismutho-Phenolic Compounds.—E. Richard. —A diatomic phenol, such as $\text{C}_6\text{H}_4(\text{OH})_2$, combines with the hydrate of bismuthyl, and gives, owing to the loss of a molecule of water, a compound answering to the following formula:—



For such a reaction to take place it is first necessary for the phenol to have at least two oxyhydriles; another no less important condition is that these two oxyhydriles must be opposite one another in the ortho-position. To prepare these compounds, it is sufficient to add a solution of a bismuth compound to a solution of phenol. For instance, if we add to a slightly warmed solution of pyrocatechin a solution containing an equal weight of bismuth emetic, the solution will first take a yellow colour, then become cloudy, and finally give an abundant deposit of a citron-yellow colour, which is shown by analysis to correspond with the rough formula $\text{C}_6\text{H}_2\text{O}_2 = \text{Bi}$, and which is, in fact, the pyrocatechin hydrate of bismuthyl, $\text{C}_6\text{H}_4\text{O}_2 = \text{Bi} - \text{OH}$, insoluble in water, alcohol, and chloroform. It is decomposed by sulphuric acid, giving sulphate of bismuth; the pyrocatechin is set free, and can be separated with ether. It is decomposed with violence by nitric acid. If, instead of using pyrocatechin, we use resorcin or hydroquinone, no similar reaction takes place, but, on the other hand, this easily occurs if the other diphenols with two oxyhydriles in ortho are used. Gallic acid and gallate of methyl also form a compound with bismuth under the same conditions.

Diphenylcarbazide, a very Sensitive Reagent for some Metallic Compounds (Copper, Mercury, Iron, Chromic Acid).—P. Caseneuve.—Already noticed.

No. 5.

This number contains no matter of chemical interest.

No. 6.

Analysis of the Waters from the Hot Springs at Djebel Achkel.—M. Puaux.—Already inserted in full.

The Presence of Zinc in Certain Alcohols.—Th. Roman and G. Delluc.—The residue from the evaporation of 1 litre of alcohol at 95° was treated with water, slightly acidulated with acetic acid, and the whole boiled to facilitate the solution and drive off the excess of acid. After filtering, soda gave a white precipitate soluble in excess, carbonate of potassium a white precipitate insoluble in excess, sulphhydrate of ammonia a white precipitate, and ferricyanide a flesh coloured precipitate. The precipitate obtained with carbonate of potassium left, on calcination, a residue which was yellow when hot and white when cold. The presence of zinc is no doubt due to the nature of the vessels in which the alcohol was kept: this was proved by leaving some scrapings from a galvanised iron drum in contact with alcohol at 95° for two days, with frequent agitation, when the presence of zinc in solution was detected; after four days' contact the indications were very strong. The zinc was detected by the addition of urobiline, which gives a greenish fluorescence: this substance is best added in the form of a chloroformic solution of urobiline, which is easily prepared.

Glycerophosphate of Quinine.—M. Prunier.—This substance is best prepared in the following manner:—A saturated solution of glycerophosphate of lime is gradually added to a solution of oxalic acid at about one-twentieth, and carefully stirred, a slight excess of the glycerophosphate must be finally added; this is left for some hours before filtering, so as to precipitate the whole of the oxalic acid. To the clear filtrate a slight excess of hydrate of quinine, suspended in water, is gradually added; this is left until it gives an alkaline reaction with litmus. After saturation in the cold, the whole is brought to boiling to dissolve the salt, and to separate the excess of insoluble quinine by hot filtration. On cooling, an abundant crop of crystals of basic glycerophosphate of quinine is obtained. Under these conditions the salt contains 5 molecules of water, which it gradually loses when the temperature is raised; above 60° it turns brown, and commences to decompose. Its composition is shown to be—Quinine 70 parts, glycerophosphoric acid 19 parts, water 11 parts.

MEETINGS FOR THE WEEK.

TUESDAY, Jan. 1st. (Royal Institution, 3. (Christmas Lectures to Young People). "On Great Chapters from the Book of Nature," by Sir Robert Stawell Ball, D.Sc., LL.D., F.R.S.
THURSDAY, " 3rd.
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Further particulars on application to the Director—
ALFRED JÖRGENSEN, The Laboratory, Copenhagen, V.

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" " do. No. 3 " 12° C.	
" " do. No. 4 " 0° C.	

CARBOLIC ACID DISINFECTING POWDER.

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